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Pilot Plant Testing of Caustic Spray/Hot Gas Building Decontamination Process

(Task Order Number 5)

Final Report

A.A. Balasco
Program Manager

L.R. Woodland — Task Leader
J.W. Adams
K.J. Beitis
D.E. Kleinschmidt
M.A. Randel
M. Rossetti
K.E. Thrun
Principal Investigators

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The U.S. Army operates and/or owns ammunition plants and depots involved in the manufacture, processing, loading and storage of pyrotechnic, explosives and propellant (PEP) materials. These operations involve permanent facilities and a variety of process and handling equipment. Many of these facilities are in an inactive or stand-by status and are candidates for excessing operations. In some cases, explosive contaminated structures have significant reuse potential for conversion to other industrial processes. In order to recover these valuable resources, non-destructive decontamination techniques are necessary to eliminate the explosive and toxic hazard of any munition processing wastes.

The use of hot gas treatment (with or without caustic spray) appears to be a promising means of decontaminating explosive contaminated buildings. More importantly, this hot

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1.0 SUMMARY

The use of hot gas treatment (with or without caustic spray) appears to be a promising means of decontaminating explosive contaminated buildings. More importantly, this hot gas decontamination process appears to be non-destructive in nature and, therefore, has the potential for rendering treated buildings available for reuse or excessing operations.

The caustic spray/hot gas building decontamination system used during our pilot tests at Cornhusker Army Ammunition Plant (AAP), located in Grand Island, NE, consisted of two burners and a remote control station. An air heater supplied hot gas to the test areas and an afterburner incinerated the organics in the hot gas leaving the test area. In nearly all respects, our pilot testing achieved on a much larger scale (with an actual building) that which was previously achieved in the laboratory. This pilot-scale testing (in two 11 ft x 24 ft x 12 ft ht. test areas of the explosive contaminated building) indicates that a hot gas treatment using a 900°F gas stream to heat the inside wall and floor surfaces to about 500°F will:

- Reduce the wall (or floor) surface explosive contaminant (TNT) concentration to about 1 ug/sq cm;

- Reduce the (concrete block) wall interior explosive contaminant (TNT) concentration to about 0.11 ug/gm (at the detectable limit) of wall material; and

- Minimize the loss in structural strength of (concrete block) walls; 5% loss in compressive strength and 20 to 30% loss in bend (tensile) strength.

As indicated above, the residual interior explosive contaminant (TNT) concentration of the concrete blocks (when heated to 500°F) was reduced to near detectable limits in our two pilot tests. In contrast, the residual surface explosive contaminant concentration of the concrete blocks generally exceeded the detectable limit (about 0.3 ug/sq cm) unless the concrete blocks' surface was heated above 600°F. Although considerable decontamination of the surface and interior of the blocks was achieved, to the best of our knowledge, there are no currently existing standards which specify what an acceptable level of residual explosive contaminant would be. In light of this, it may be difficult to judge whether this decontamination process will render such treated buildings available for excessing operations. This is a question that must eventually be resolved by the U.S. Army in order for the effectiveness of this promising technology to be evaluated.

In addition, there were a number of aspects of the decontamination process that could not have been investigated in laboratory-scale tests but were successfully proved-out in the pilot tests. These included the following:

- o Feasibility of remote operation of burner, gas handling and gas sampling systems;

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- Ability to achieve even heat distribution to the test area walls and floor;

- Gross effect f heating rate and temperature on building structural integrity (distortion);

- Measurement of the resultant temperature gradient across the walls and floor (for assessment of structural effects and DRE);

- Measurement of heat transfer rate and estimation of heat losses;

- Feasibility of push-pull pressure control within the test building; and

- Measurement and effectiveness of control of gaseous emissions from the building test area.

Remote operation capability for both the burner and gas sampling systems may be critical safety considerations where high levels of explosive are known or suspected to exist. The pilot testing at Cornhusker AAP has shown that remote operation of the burner systems and gas sampling is practical.

With a minimum of gas flow correction (a simple steel sheet gas flow baffle) it was possible to achieve a wall temperature that varied only about ± 40°F from the average. Noting the location of the "cold" spots in the first pilot test, it is apparent that some additional openings in the back (north) side of the inlet duct could have reduced this temperature differential to as little as ± 20°F from the average. (By monitoring the temperature profile in future pilot-scale or full-scale testing, it should be possible to develop modifications in the gas flow patterns to limit this temperature variability to about ± 20 to 25°F for full-scale applications.)

Visually observable structural damage (formation or widening of cracks in the walls or floor) is affected by heatup rate. An inlet g ' temperature increase of about 50°F per hour caused no visible structural damage in the first pilot test, whereas visible damage did occur when the inlet gas temperature was increased 130 to 200°F per hour in the second pilot test. This visible structural damage in the second pilot test included the formation of new cracks in the concrete floor and concrete block walls as well as widening of existing cracks in the walls. In addition to the more rapid heat up rate(s) in the second pilot test, there were three heat up/cool down cycles compared with only one in the first pilot test. The cement block cool down rates in the second pilot test were approximately the same, however, as the cool down rate in the first pilot test (150°F the first hour, 50°F the second hour). Although either the thermal cycling or the more rapid heat up rate may have caused the structural damage in the second pilot test, it seems more likely to have been the greater thermal stress in the rapid heating. Thus, it is recommended that the inlet gas temperature be limited to an increase of about 50°F per hour.

Minimizing the temperature gradient across the wall is important for two reasons. It reduces heat losses and, therefore, heat input requirements and it minimizes the inner surface temperature necessary to achieve a given

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outside surface temperature (overall average wall temperature). The temperature gradient across the outside wall in our first pilot test (for areas with good hot gas circulation) was about 100°F with 6 inches of mineral wool insulation [k = 0.02 Btu/(hr)(sq ft) (°F/ft)]. This appears to be the minimum acceptable degree of insulation because less insulation would increase the temperature gradient across the wall and require a higher (more structurally damaging) temperature for the inner wall surface. Rigid, reusable, insulating board would probably be a more practical type of insulation for full-scale application of this process.

The pilot testing also allowed the heat transfer rate to the building test area and heat losses to actually be measured as opposed to theoretically estimated, thus allowing a more accurate estimate to be made of heat input requirements and rates for full-scale application of the process.

"Push-pull" pressure control (i.e., the use of two fans for maintaining gas flow and pressure) of the building area being tested can sometimes present an unstable condition where one or both fans or fan dampers oscillate if the control system is not properly designed. This was taken into consideration in the design of our pilot plant gas handling system and proved both feasible and easily maintainable. Although it was originally anticipated that the test area would be operated at 0 to -0.5 in W.C. to prevent the release of contaminated gases, the pilot control system was later modified to operate over a much narrower pressure range (-0.01 to -0.02 in W.C.) to minimize air inleakage. Although this single pressure sensor was perfectly adequate for a small building, two or more pressure sensors may be required for larger structures, particularly where there is variation in the cross section of the building that could cause a measurable pressure drop due to gas flow. The control system should be operated, using the pressure sensor showing the highest absolute pressure, to maintain whatever negative pressure is necessary to prevent building pressure from ever going positive, even under high wind conditions. During high wind conditions in our pilot tests, it was necessary to operate at -0.02 in. W.C. to always maintain a negative building pressure, whereas the building could be operated at -0.01 in. W.C. during no wind conditions.

The effectiveness of an afterburner in treating the vapors in the contaminated gas stream from the treated building test area has been evaluated. Destruction efficiency (DRE) of the very dilute explosive (TNT) vapors from the treated area, as measured across the afterburner for a one-second retention time at 1800°F, was 99.7%; slightly below the 99.99% required by the Environmental Protection Agency (EPA) for incineration of hazardous waste. The lower DRE is principally the result of an atypically low inlet concentration of the contaminant. This destruction efficiency resulted in a residual level of TNT of less than one ppb in the afterburner outlet which may still be allowable under ambient air regulations. Concentrations of other regulated principal organic hazardous constituents (POHCs) in the afterburner outlet gas (such as chrysene) were also only in the ppb range. In addition, particulate concentration (at about 0.00014 gr/scfd) in the afterburner stack gas, without the use of any particulate control device, is well below the 0.08 gr/scfd required by EPA regulations. Consequently, we do not see the need for particulate control.

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In summary, it appears this hot gas decontamination process for treating explosive contaminated structures is very promising and warrants further investigation. For example, additional laboratory and/or pilot-scale testing on a number of other building materials and building configurations would prove extremely useful. (This might include testing on buildings and/or equipment currently requiring decontamination.)

This additional testing should include determining: (a) the effectiveness of this decontamination process with other explosives; (b) the time/temperature relationship to decontamination efficiency; and (c) the effect of time and temperature on the materials of construction.

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2.0 BACKGROUND

The U.S. Army operates and/or owns ammunition plants and depots involved in the manufacture, processing, loading and storage of pyrotechnic, explosives and propellant (PEP) materials. These operations involve permanent facilities and a variety of process and handling equipment. Many of these facilities are in an inactive or stand-by status and are candidates for excessing operations. In some cases, explosive contaminated structures have significant reuse potential for conversion to other industrial processes. In order to recover these valuable resources, non-destructive decontamination techniques are necessary to eliminate the explosive and toxic hazard of any munition processing wastes. Furthermore, other such facilities have been condemned or slated for demolition and a safe and effective treatment method is needed to remove or destroy hazardous contaminants prior to disposal of the rubble.

This task order [Task Order Number 5 under U.S. Army Toxic and Hazardous Materials Agency (USATHAMA) Contract No. DAAK 11-85-D-0008] constitutes Phase III of the Development of Novel Decontamination Techniques for Explosives Contaminated Facilities. In the Phase I Concept Development effort [performed by Battelle Columbus Laboratories (BCL)], ideas were generated and specific concept treatment systems were formulated.[1] The concepts were evaluated against technical, safety and economic criteria. Five concepts were selected for Phase II Laboratory Development: hot gas, solvent vapor condensation, solvent extraction, chemical reaction (radical initiated, base initiated and sulfur based reductions) and combined chemical/hot gas.[2] In the laboratory development program (also performed by BCL), each technique was evaluated for decontamination effectiveness on painted and unpainted steel and painted and unpainted concrete. Liquid and gaseous effluents were analyzed for explosives and decomposition products of explosives. Based on the laboratory results, a combined caustic treatment/exhaust gas concept was selected for pilot development (Phase III).

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[1]Benecke, et al., "Development of Novel Decontaminating and Inerting Techniques for Explosives Contaminated Facilities, Phase I Identification and Evaluation of Concepts," Report No. DRXTH-TE-CR-83211 to USATHAMA under Contract No. DAAK-11-81-C-0101 by Battelle Columbus Laboratories (July 1983).

[2]Hooper, et al., "Development of Novel Decontamination and Inerting Techniques for Explosive Contaminated Facilities, Laboratory Evaluation of Concepts," Report No. AMXTH-TE-TR-85009 to USATHAMA under Contract No. DAAK-11-81-C-0101 by Battelle Columbus Laboratories (March 1, 1985).

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3.0 OBJECTIVES

The objectives of this Phase III caustic spray/hot gas building decontamination pilot plant test program were fourfold:

(1)   Determine the effectiveness of decontamination using hot gas with and without caustic spray on an actual explosive contaminated structure;

(2)   Evaluate the effects of these test conditions on the integrity of an actual structure;

(3)   Provide preliminary design criteria information for a full-scale system design; and

(4)   Provide test data for regulatory permitting of this process.

4.0 SELECTION OF TEST PARAMETERS

As stated in Sections 2.0 and 3.0, the main objectives of this pilot test program were to determine the effects of heat and heat plus chemical decontamination on an actual explosive contaminated structure, both in terms of decontamination efficiency and structural integrity.

In the preceding laboratory test program conducted by BCL, a number of explosives, construction materials, decontaminating solutions and test temperatures were investigated. From those tests it was determined that heating metal or concrete surfaces with hot gas (with no chemical pretreatment) to a temperature of 500°F for one hour was sufficient to either vaporize or decompose all of the different explosives tested. Removal efficiencies of 99.9 to 99.99% were obtained. The explosives tested by this procedure included the following:

- 2,4,6-Trinitrotuluene (TNT),

- Cyclotrimethylenetrinitramine (RDX),

- Homocyclonite (HMX),

- 2,4,6-Trinitrophenylmethylnitramine (Tetryl),

- 2,4-Dinitrotoluene (DNT), and

- 2,6-Dinitrotoluene (DNT).

The chemical decontamination concept explored in the laboratory study indicated that a four-hour treatment with a solution of 0.1N sodium hydroxide (NaOH) in co-solvent dimethylsulfoxide (DMSO) would destroy residual explosive in efficiencies of 99% on steel or 70 to 99.99% on concrete (99.99% for TNT). A combination treatment of four hours chemical treatment plus one hour of hot gas treatment at 300°F was found to remove even the more resistant explosive (2,6 DNT) from the concrete upons in efficiencies of greater than 99%.

The chemical spray solution recommended for decontamination was 0.1N NaOH solution containing a co-solvent (at 30 to 75% by wt.) to solubilize the explosive. Three co-solvents appeared most promising: acetone, dimethylsulfoxide (DMSO) and dimethylformamide (DMF). Acetone was very effective, but was considered too flammable and was eliminated from further consideration by BCL in favor of DMSO and DMF.

In subsequent discussions between Arthur D. Little, Inc. and USATHAMA personnel, it was decided that DMF would be a better choice for a co-solvent for two reasons. First, DMSO could have potential side effects on personnel exposed during the spray application and in the hot gas treatment step, and secondly sulfur dioxide (SO_2) would be generated from incineration of the vapors from the DMSO in the spray solution. DMF may be slightly less effective than DMSO as a co-solvent, but this is greatly outweighed by the potential toxicity and the SO_2 pollution abatement requirement if DMSO were used.

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Following discussions with USATHAMA, the following conditions were then selected for pilot-scale testing:

- Hot gas treatment alone at 500 to 750°F for one hour (500°F minimum exterior building surface and 750°F maximum interior building surface temperatures),

- Application of chemical decontaminating spray for one hour followed by one hour of hot gas treatment at 500 to 750°F, and

- Chemical decontaminating spray composition of 30% by weight DMF, 4 grams NaOH per liter of solution, and the balance water.

5.0 EQUIPMENT AND FACILITIES

5.1 Building Test Site Selection

Six different facility areas at Cornhusker AAP were examined as potential test sites for pilot plant testing. These six sites were first evaluated on the basis of their materials of construction and accessibility for testing and then the number of potential test sites was narrowed to three. These three candidate sites were then photographed using visible and ultraviolet (UV) light to detect the presence of explosive contamination. Wipe tests and chemical analysis were next used to confirm the presence and determine the concentration of explosive contamination. Building 4L-5 at Cornhusker AAP was finally selected as the test site for this pilot plant program.[3]

As the result of this testing to determine initial surface contamination, none of the buildings (or test sites) were found to be sufficiently contaminated for testing as is. As described more fully in the Field Survey/Selection Report (Balasco, et al.), even in Building 4L-5 only eight surface areas had quantifiable quantities (>25 ug/filter by HPLC analysis) of contamination. These few areas of measurable contamination were too small in size (1 to 2 inches across) to allow replicate sampling for testing purposes (sampling before and after treatment). Contaminated concrete blocks excavated from the sump cesspool just outside Building 4L-5 were found to be sufficiently contaminated with TNT to provide replicate samples of explosive contaminated surfaces. A concrete block from row 8 of the sump cesspool, for example, showed a surface explosive (TNT) concentration of 400 times the average level of contamination of the eight samples with the highest TNT concentration taken in Building 4L-5. In addition, the evenness of coloration across the face of each row of cesspool concrete blocks initially indicated that contamination of these blocks might be uniform enough for multiple, reproducible sampling.

Consequently, it was decided that contaminated cesspool blocks would, therefore, be inserted in two test walls to be constructed in Building 4L-5 to carry out the decontamination testing; one in the west test area and one in the east test area. (See Figures 5-4, 5-5 and 5-6 of Section 5.3 for the specific locations of these test blocks.)

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[3]For more details of the test site selection and the methods used for measuring the levels of explosive contamination refer to Balasco, et al., "Field Survey/Selection of Pilot Test Site for Building Decontamination Test Program," Report to USATHAMA under Contract No. DAAK11-85-D-0008 by Arthur D. Little, Inc. (December 1985).

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5.2 Building Modifications

Prior to starting any of the building modifications (to Building 4L-5) or installation of test equipment a Test Plan[4] and Safety Plan[5] were prepared. The Test Plan was prepared by Arthur D. Little, Inc., while the Safety Plan (A Preliminary Hazards Analysis or PHA) was prepared by Hercules Aerospace Co. (RAAP) and included a review of the proposed Test Plan. After the Test Plan was revised to reflect (and correct) the safety issues discussed in the PHA, it was approved by USATHAMA. Following approval of the Test Plan, building modifications and equipment installation was begun.

A number of modifications were performed by Mason & Hanger-Silas Mason Co., Inc. (government contractor maintaining Cornhusker AAP in its stand-by status) to prepare Building 4L-5 for the pilot-scale decontamination testing. These included the following:

- Removal of the existing 8-inch projectile washout machine, piping, wiring, overhead crane, lights and wooden window frames;

- Replacement of windows and doors with sheet steel;

- Construction of a dividing wall down the center of Building 4L-5 to provide two test areas and allow for the installation of explosive contaminated concrete blocks (from Building 4L-5 sump cesspool) for the first pilot test (in the west side of the building);

- Construction of a false ceiling of 1/8th inch sheet steel in Building 4L-5 just above window height to reduce the test area volume and protect the peaked wood roof;

- Installation of six inches of high temperature insulation on the exterior of Building 4L-5, on top of the false ceiling, and in the center wall, to reduce heat losses from the test areas and minimize the temperature gradient across the concrete block walls;

- Installation and insulation of duct work to provide hot air to the building from the air heater and convey the contaminated air from the building to the afterburner;

- Construction of sheet steel hot air deflectors in the two test areas to direct hot air to the floor; and

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[4]Balasco, et al., Pilot Plant Testing of Caustic Spray/Hot Gas Building Decontaminatin Process--Test Plan" Report to USATHAMA under Contract No. DAAK11-85-D-0008 by Arthur D. Little, Inc. (May 1986).

[5]Cabbage, W., "Memorandum Report--Preliminary Hazards Analysis of Caustic Spray/Hot Gas Decontamination Pilot Test Program," HI-85-M-91, Report to USATHAMA under Contract No. DAAK11-85-D-0008 by Hercules Aerospace Company (RAAP) for Arthur D. Little, Inc. (December 1985).

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• Construction of a concrete block baffle wall in the east half of Building 4L-5 at the end of the hot air deflector to prevent short circuiting of the air flow through the room and allow for the installation of explosive contaminated concrete blocks (from Building 4L-5 sump cesspool) for the second pilot test.

5.3 Equipment Layout and Thermocouple Placement

Figure 5-1 is a photograph of the two burner (air heater and afterburner) systems and Building 4L-5 itself, while Figure 5-2 presents a schematic of the two burner systems and Building 4L-5. The east and west test areas of Building 4L-5 both measured approximately 11 ft x 24 ft x 12 ft height. (For further information concerning dimensions and/or distances, refer to Figure 5-2.)

To monitor the inside and outside temperature of the structural walls, floor and contaminated test blocks during heat up (and cool down), thermocouples (a total of 36 in Pilot Test 1 and 38 in the second pilot test) were installed on the inside and outside surfaces of the concrete blocks using Saureisen concrete. Two additional thermocouples were installed as a safety precaution inside the peak of the wooden roof of Building 4L-5 to sense any abnormal temperature rise that might indicate the presence of a fire. The location of all thermocouples employed in both test areas is shown in Figures 5-3 through 5-6. Figure 5-3 indicates the location of the thermocouples in the west wall for structural test monitoring during the first pilot test. Likewise, thermocouple location in the contaminated test blocks in west side of dividing wall used in the first pilot test is presented in Figure 5-4. Figure 5-5 shows thermocouple locations in both the east structural wall and floor, placement of the hot air deflector, and the concrete block baffle wall in the east test area of Building 4L-5 used in the second pilot test. Figure 5-6 indicates the locations of the thermocouples in the contaminated test blocks in the concrete block baffle wall used in the second pilot test.

5.4 Burner Design and Control

5.4.1 Burner Design

Both burners (building air heater and afterburner) were designed to operate on propane fuel and each were equipped with their own air handling fan rated at 2000 scfm. The air heater supplied the hot air (flue gas) to heat the test area. The afterburner (thermal oxidizer) incinerated the air exiting from the test area to destroy any vapors of explosive or explosive decomposition products present in the contaminated air.

The general specifications for the two burner systems were as follows:

Building Air Heater

- Propane fired
- 1000 to 2000 scfm at 500 to 1000°F
- Maximum of 3 million Btu/hr gross input

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FIGURE 5-1 CAUSTIC SPRAY/HOT GAS BUILDING DECONTAMINATION TEST SITE
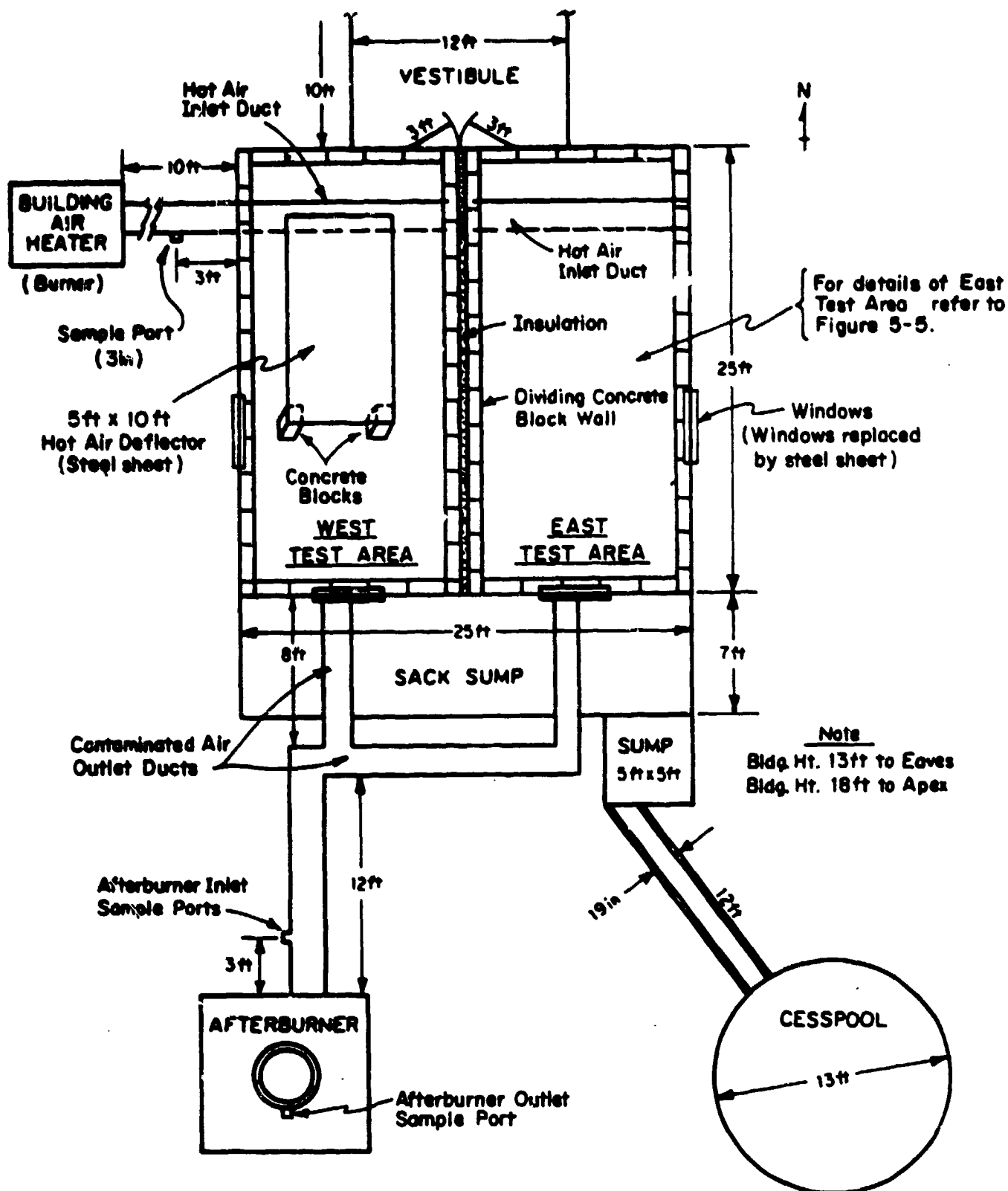
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FIGURE 5-2 SCHEMATIC OF BUILDING 4L- 5 AND DECONTAMINATION EQUIPMENT FOR THE TWO PILOT TESTS

Source: Arthur D. Little, Inc.

FIGURE 5-3 THERMOCOUPLE PLACEMENT IN BUILDING 4L-5 WEST WALL FOR STRUCTURAL TEST MONITORING – PILOT TEST 1

Source: Arthur D. Little, Inc.

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CORE SAMPLING OF CONTAMINATED BLOCKS AFTER TEST

BLOCKS A-D   BLOCKS E-H

Core Sample Either Side of Holes   Core Sample (2 pieces) Center

Outside Wall [image] Inside Wall   Outside Wall [image] Inside Wall

Caustic Spray Treat 6 Center
Test Blocks

Wall Location of Test Concrete Blocks
and Thermocouples
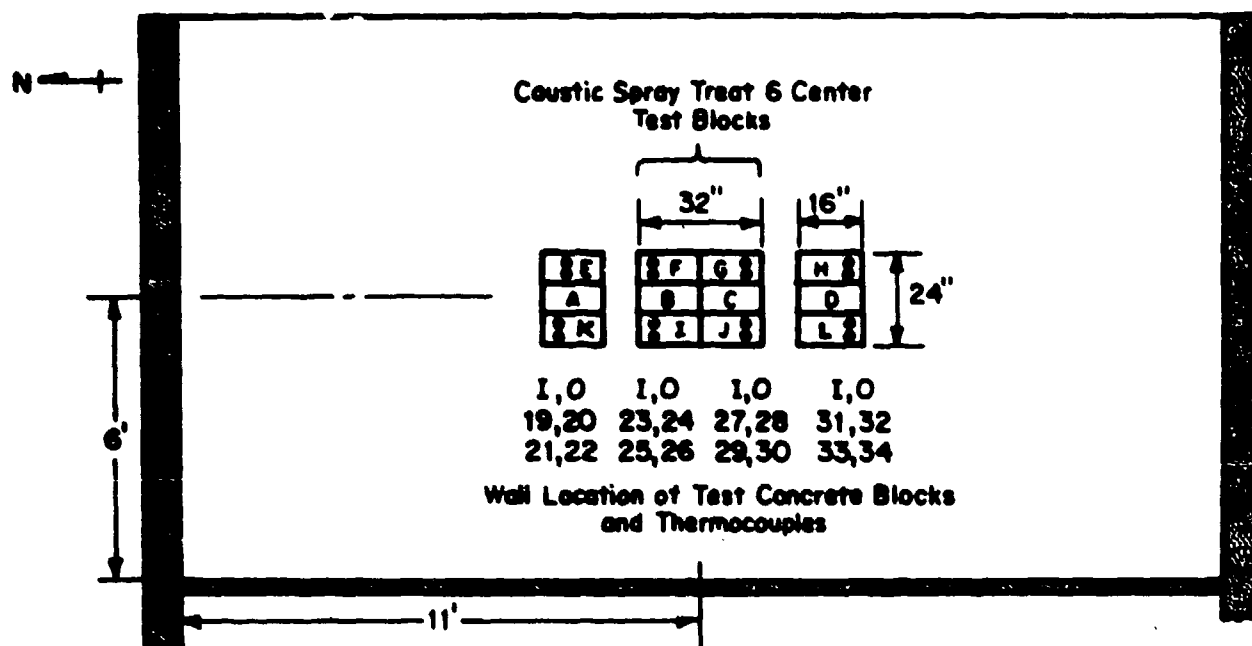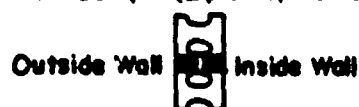
Example of Concrete Block
Thermocouple Numbering:

#21 I
#22 O
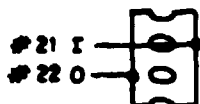
I = INSIDE SURFACE
O = OUTSIDE SURFACE

Note: All explosive contaminated
      test blocks from sump cesspool

FIGURE 5-4   LOCATION OF TEST CONTAMINATED CONCRETE BLOCKS
AND THERMOCOUPLES IN WEST SIDE OF BUILDING
4L-5 DIVIDING WALL - PILOT TEST 1

Source: Arthur D. Little, Inc.

Arthur D. Little, Inc.   5-7
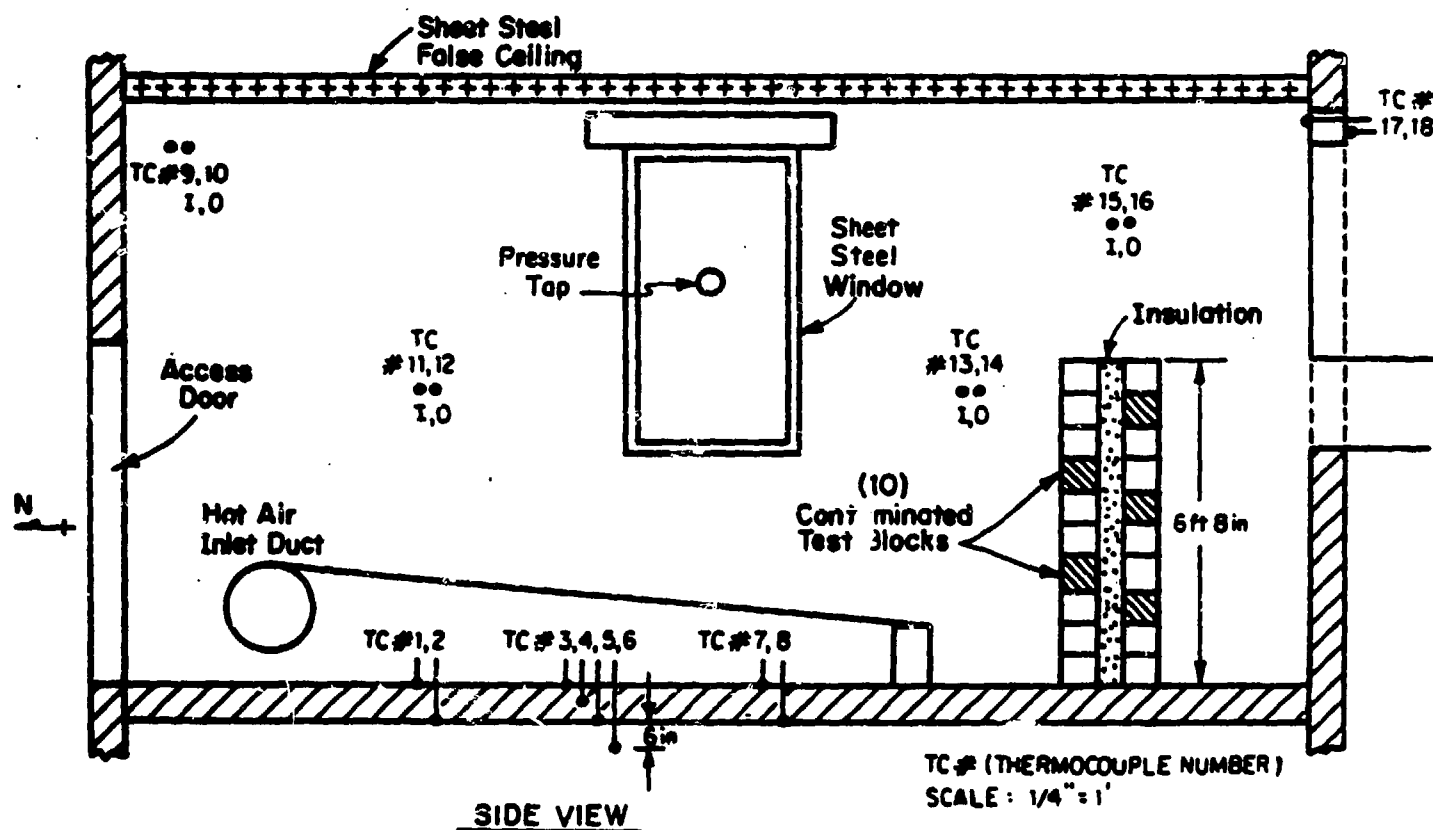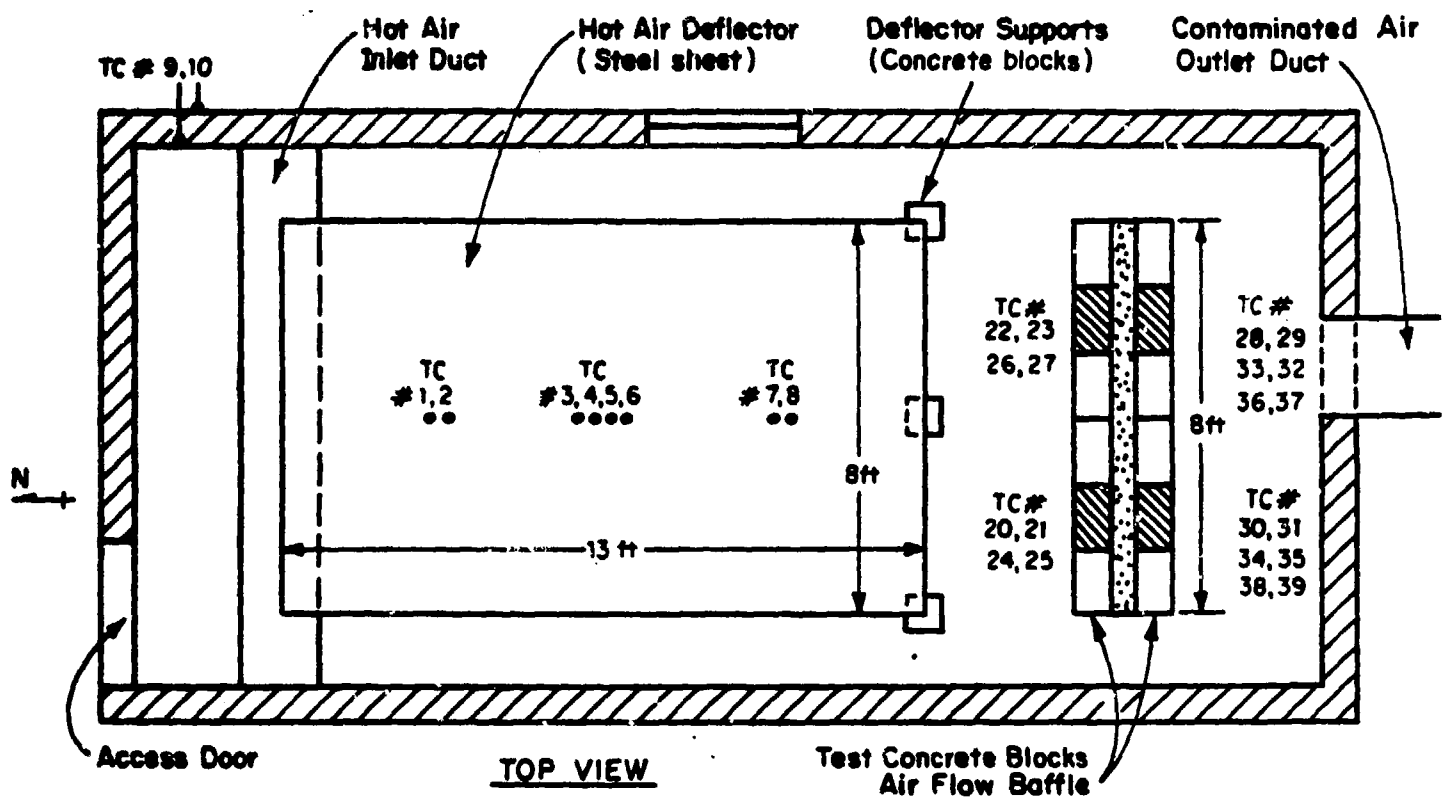
FIGURE 5-5 THERMOCOUPLE PLACEMENT IN BUILDING 4L-5 EAST WALL – PILOT TEST 2

Source: Arthur D. Little, Inc.

TEST BLOCKS AND THERMOCOUPLES (Facing south wall from north wall)

THERMOCOUPLES	BLOCKS		BLOCKS	THERMOCOUPLES	
22 23	7-30		8-7	20 21	
I O				I O	
26 27	7-2		8-27	24 25	
I O				I O	

TEST BLOCKS AND THERMOCOUPLES (Facing north wall from south wall)

THERMOCOUPLES	BLOCKS		BLOCKS	THERMOCOUPLES	
30 31	7-29		7-20	28 29	
I O				I O	
34 35	8-10A	Caustic Sprayed	7-8	33 32	
I O				I O	
38 39	8-10B		7-9	36 37	
I O				I O	

Key: I = Inside Surface (toward room)
     O = Outside Block (toward insulation)

FIGURE 5-6   LOCATION OF THERMOCOUPLES IN TEST CONTAMINATED
             CONCRETE BLOCKS — PILOT TEST 2

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Afterburner (Thermal Oxidizer)

- Propane fired
- 2 second residence time at 2000 scfm
- Maximum of 2000°F operating temperature
- Maximum of 4.5 million Btu/hr gross input

5.4.2 Burner Control

Because of the potential for the presence of undetected explosives in the soil under and around the test site (Building 4L-5), the control panel for the two burner systems was located approximately 520 ft. from the building in a mobile trailer shielded by an earth embankment. Actual sampling of soil from beneath Building 4L-5 had indicated explosive levels of less than 1 ppm, but the area that could be sampled was limited.

The site plan for the burner systems, propane fuel tanks and mobile control trailer is presented in Figure 5-7. The embankment shielded the control trailer and operating personnel during the testing. The propane tanks were located about 100 ft from the burner systems, as recommended in the hazards analysis, to protect the tanks in case of malfunction of the burners as well as to protect the buildings in case of a propane tank leak, fire or explosion.

Additional test and data recording equipment was located behind the blast door (Figure 5-7) in the corridor leading to Building 4L-5. A data logging type recorder recorded all the floor, wall, roof, and contaminated concrete block temperatures in the test areas as measured by the thermocouples during both pilot-scale tests. A Beckman 402 Flame Ionization Hydrocarbon Analyzer was also located in this area behind the blast door to monitor hydrocarbon concentrations in the air leaving Building 4L-5 (and entering the afterburner) and transmit this information to the control trailer.

During the actual testing, personnel periodically checked this equipment behind the blast door by moving from the control trailer to the other side of Line 4, behind Building L-10 (Figure 5-7), using a route shielded by embankments and blast containment structures.

With respect to system safety, standard industrial safety features built into the two burner systems included:

- Automatic shutdown of burners if flameout was sensed by a UV flame detector;

- Automatic shutdown of burners if high or low gas pressure or high or low burner temperature was sensed; and

- Automatic shutdown of burners if loss of air flow through the afterburner fan was detected.

In addition to the typical safeguards which were built into this system design, two other automatic shutdown capabilities were deemed necessary. These were:

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FIGURE 5-7   SITE PLAN FOR DECONTAMINATION OF BUILDING 4L-5

Source: Mason & Hanger-Silas Mason Co., Inc.
Cornhusker Army Ammunition Plant

- Burner (air heater) shutdown and fan damper closure in the case of a high combustible level in air exiting from Building 4L-5; and

- Entire-system shutdown in the case of high (greater than +1.0 in W.C.) or low (less than -1.0 in W.C.) building pressure.

The Beckman 402 Hydrocarbon Analyzer which was used to monitor the hydrocarbon level in the hot gases leaving Building 4L-5 and entering the afterburner was also tied into the automatic shutdown system. In the event this gas stream reached the lower explosive limit (LEL) in vaporized organics such as explosive, DMF solvent, or paint pyrolysis products an automatic shutdown of the entire system would occur.

The building pressure controller operated the afterburner fan damper in order to hold the building test area at a slightly negative pressure (-0.02 in W.C.) thereby keeping hot (contaminated) air from leaking out of the building. The high and low pressure automatic shutdown switches were installed to prevent extreme negative or positive pressure damage such as collapse of the walls or false ceiling. (In the checkout of the air handling system, it was found that because of the high air leakage rate it was not possible to achieve greater than a 1.0 in W.C. pressure differential--either positive or negative, so there was no possibility of building damage due to pressure differential.) It as originally anticipated that the building would be operated ai 0.5 in W.C. pressure, but air flow measurements indicated that this would result in too much air in-leakage. While the air handling system (fans and dampers) might have been able to handle the additional air in-leakage, it would not have been possible to reach 2000°F in the afterburner. Therefore, the test areas were operated at a minimum negative pressure of from -0.01 to -0.02 in W.C. during the pilot tests.

A process and instrument diagram for both burner systems and controls is shown as Figure 5-8. Table 5-1 lists the instruments depicted in Figure 5-8.

5.4.3 Emergency Shutdown of System

In addition to the automatic shutdown safety features of the system, there was also a "panic button" on the control panel that could be used in the event of an unanticipated problem arising that would not automatically initiate shutdown of the system.

During the second pilot test it was necessary to use this "panic button" to shut down the system when the plywood holding the insulation around the outside of the building became heated and ignited. As a safety precaution, firehoses had previously been laid out around and inside the corridor adjoining Building 4L-5 before the start of the testing and these hoses were used to quickly extinguish the small fire. When the building had cooled sufficiently and the burned plywood had been cut away, the test was restarted.
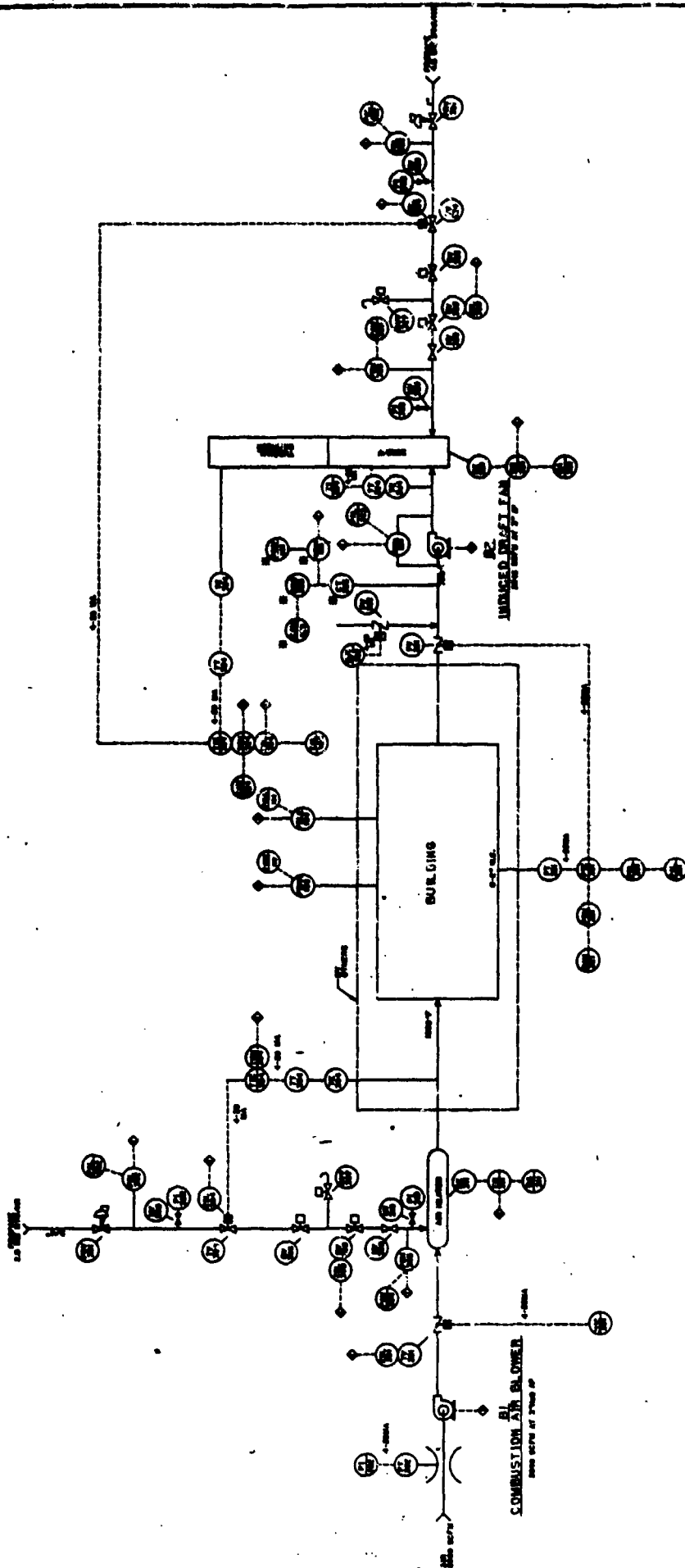
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FIGURE 5-6 BURNER SYSTEM PROCESS AND INSTRUMENT DIAGRAM

5-13

TABLE 5-1

INSTRUMENT LIST FOR HOT GAS DECONTAMINATION BURNER SYSTEMS

A. **AIR HEATER**

PT-202	Pressure Transmitter
PI-202	Pressure Indicator
ZSL-201	Damper Position Switch
HIC-201	(Remote) Manual Damper Positioner
TV-201	Damper Positioner
PCV-301	Propane Pressure Regulator
PSL-302	Propane Pressure Switch Low
PI-303	Propane Pressure Indicator
TV-304	Temperature (Propane Flow) Valve
ZSL-304	Valve Position Switch
PSH-305	Propane Pressure Switch High
BV-306	Propane Block Valve
BVV-307	Propane Vent Valve
BV-308	Propane Block Valve
HV-309	Hand Valve
PI-310	Propane Pressure Indicator
TE-304	Temperature Element
TT-304	Temperature Transmitter
TIC-304	Temperature Indicating Controller
TSH-304	Temperature Switch High
ZSL-308	Valve Position Switch
BE-311	Burner Eye (Flame Sensor)

B. **BUILDING**

PT-221	Building Pressure Transmitter
PIC-221	Pressure Indicating Controller
PAH-221	Pressure Alarm High
PV-221	Afterburner Damper
HV-222	Bypass Damper
PDSL-223	Pressure Differential Switch Low
TI-224	Building Outlet Gas Temperature Indicator
PAL-221	Pressure Alarm Low
PSHH	Pressure Switch High High (Shutdown)
PSLL	Pressure Switch Low Low (Shutdown)

TABLE 5-1 (Continued)

INSTRUMENT LIST FOR HOT GAS DECONTAMINATION BURNER SYSTEMS

C.   THERMAL OXIDIZER (AFTERBURNER)

TE-324	Afterburner Temperature Element
TT-324	Temperature Transmitter
TIC-324	Temperature Indicating Controller
TSH-324	Temperature Switch High
TSL-324	Temperature Switch Low
CT-225	Combustibles Transmitter
CAHH-225	Combustibles Switch High High
CAH-225	Combustibles Switch High
PRV-321	Propane Pressure Regulator
PSL-322	Propane Pressure Switch Low
PI-323	Propane Pressure Indicator
TV-324	Temperature (Propane Flow) Valve
PSH-325	Propane Pressure Switch High
BV-326	Propane Block Valve
BVV-327	Propane Vent Valve
BV-328	Propane Block Valve
HV-329	Hand Valve
PI-330	Propane Pressure Indicator
ZSL-328	Valve Position Switch
PDSL-223	Fan Pressure Switch
BE-331	Burner Eye (Flame Sensor)

Source: John Zink Company

6.0 PREPARATION FOR PILOT TESTING

6.1 Air Flow Measurement

Prior to the pilot-scale testing, the two burner systems (and Building 4L-5 test area) were operated with cold air to: (1) allow calibration of the air flow meter on the air heater; (2) to measure air flow through the after-burner and estimate air in-leakage to the test area; and (3) to verify correct operation of the test area pressure control instrumentation. A pitot tube was used during this check-out procedure to traverse the inlet and outlet ducts of the test area to measure air flow from the air heater and to the afterburner.

While operating the two burner systems on cold air, the afterburner fan motor tripped repeatedly. By taking afterburner fan motor amperage readings during the pitot tube measurement of airflow into the inlet duct of the afterburner for various fan damper settings, it was determined that the 5 HP fan motor was drawing more amperage than it should for the specified flow rate. After discussions with the fan supplier, the 5 HP motor was replaced with a 7.5 HP motor which worked reliably throughout the rest of the system checkout and testing.

As indicated in Section 5.4.2, there was so much air leakage into the test area (primarily through the expansion joints in the suspended sheet steel false ceiling), that it was not possible to either under or over pressurize the test area to the extent that damage would occur to the building or false ceiling. To minimize this air in-leakage, however, the test area was operated at a minimum negative pressure of -0.01 to -0.02 in W.C. during the two pilot tests.

6.2 Instrument Checkout

Calibration of the test area pressure automatic shutdown switches and pressure control transmitter was carried out using a water manometer and milliamp (ma) signal generator. The inlet air flow venturi on the air heater was then calibrated using these same instruments in conjunction with pitot tube air flow measurements in the air heater outlet duct (test area inlet duct).

The temperature transmitters in the air heater, afterburner and the test area outlet duct were next calibrated using millivolt (mv) and milliamp (ma) signal generators.

Finally, using a ma signal from each of the transmitters, all the indicator/controller readouts in the control trailer control panel were standardized to give the correct readouts.

The day prior to the first scheduled pilot test, however, a lightning strike in the vicinity of the burner systems burned out circuit boards in the air heater and thermal oxidizer temperature transmitters and disabled the gas valve positioning controller (temperature control) of the air heater. In the first pilot test it was, therefore, necessary to operate

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the system totally on manual control and use the test ma generator to remotely operate the air heater temperature control gas valve. (By the time of the second pilot test, all these controls had been repaired and recalibrated.)

6.3 Burner Startup and Cureout

Startup procedures for the two burner systems (as detailed in the John Zink Co. Operating Manual[6]) call for a stepped curing cycle of the refractory lining in both the afterburner and air heater before routine operation.

The cureout cycle of the refractory in the afterburner (requiring about 40 hours) was carried out to a temperature of 2000°F. The cureout schedule for the afterburner called for raising the temperature to 2100°F in the final curing step, but it was not possible to reach this temperature, even at a reduced air flow of about 1,000 scfm. To assure that sufficient capacity would later be available for the pilot testing, the size of the holes in the gas nozzle were enlarged about 45% prior to the pilot tests.

When the air heater burner was started to check its operation, it was found that it would operate at low fire (about 500°F) but not at increased temperature. The problem was traced to an incorrectly fabricated flame assembly which was replaced by the John Zink Company. Once the new burner assembly was installed, the refractory was cured as specified during the first pilot test run.

6.4 Structural Test Metrology Grid

A grid (12 ft x 24 ft) of horizontal and vertical lines was painted on the inside of the west structural wall of the west test area using a heat resistant paint before the first pilot test. The purpose of this (metrology) grid was to detect any gross deformation or shifting of the structural wall as the result of heating and cooling of the test area.

6.5 Wipe and Core Sampling

Wipe samples were taken from the front and back surfaces of all the contaminated test blocks before taking core samples or installing the contaminated test blocks in the center wall (first pilot test) or baffle wall (second pilot test). These wipe samples were taken using a 9 cm filter paper moistened with acetonitrile according to the procedure outlined in an earlier Arthur D. Little, Inc. report[7]. Wipe samples of these same blocks taken again after the testing were used to compare surface contamination of the blocks before and after heating.

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[6]John Zink Co. Operating and Maintenance Manual for John Zink Thermal Oxidizer System (S.O.#: TO-546229), January 1986.

[7]Ibid Footnote 3.

Core samples were also taken from the contaminated test concrete blocks
before testing by Mason & Hanger personnel using a water cooled 24 inch
carbide coring drill. (These core samples were used to measure interior
contamination of the concrete blocks.) The resulting holes were patched
with concrete which was allowed to cure overnight. These blocks were then
drilled with a ¼ inch hammer drill to accept thermocouples that were
installed in both sides of the block (facing toward and away from the test
area). Finally, these contaminated test blocks were installed in the
dividing wall (or concrete block baffle wall) and insulation packed around
the blocks. After each of the pilot tests, pieces (samples) were broken
from the front and back of each of the test blocks to analyze for residual
explosive contamination.

6.6 Chemical Spray Application

To determine the effect of the caustic spray/hot gas treatment versus hot
gas treatment alone, a number of the contaminated test blocks were sprayed
on one side (that facing the test area) prior to heating. (Six of the
twelve contaminated test blocks were sprayed in the first pilot test and
three of the ten used in the second pilot test.)

For each of the two tests, 1.5 gallons (6 liters) of spray solution was
prepared in the following manner: to 3.4 liters of water in a 2-gallon hand
sprayer was added 0.6 liter of 1N caustic solution and 2.0 liters of
dimethylformamide (DMF). The sprayer was then shaken to mix the solution.
Personnel preparing the solution wore protective clothing including lab
coat, rubber apron, goggles and rubber gloves. The solution was prepared
in a well ventilated area (outdoors) to allow dissipation of any vapors.

Once prepared, the chemical solution was sprayed on the selected concrete
blocks placed in the test area dividing wall (or concrete block baffle
wall) at a rate of about 0.15 gal/sq ft for one hour by personnel wearing
chemical goggles, an organic vapor respirator, a rubber apron and rubber
gloves. Spray not absorbed by the blocks was collected in a plastic pan
located beneath the blocks being treated and shipped back to Arthur D.
Little, Inc. for disposal. This excess spray was neutralized, stored in an
approved hazardous waste container and disposed of by a commercial waste
handler.

6-3

7.0 PILOT TESTING

7.1 Objectives of Two Pilot Test Runs

Building 4L-5 was divided into two separate test areas of equal size by construction of a concrete block dividing wall as described in Section 5.2, so that two separate pilot tests could be conducted using Building 4L-5. The first test was to gather data on the process to determine which process parameters (such as test area heat-up rate, effect of temperature, air flow pattern and contaminant destruction efficiency) needed to be investigated further in the second pilot test. The second pilot test was performed to: (1) confirm data collected in the first pilot test run; and (2) collect additional data not obtained in the first run.

For example, maximum TNT emissions as detected by the Volatile Organic Sampling Train (VOST) occurred earlier (at lower temperature) in the first pilot test than anticipated (at 7 to 10 hours into the test run with a contaminated block surface temperature of 250 to 380°F). Consequently, in the second pilot test TNT destruction efficiency (DRE) across the afterburner was determined while sampling the gas stream into and out of the afterburner over the time period (and temperature) when the TNT emissions from Building 4L-5 were expected (based on the first pilot test) to be at maximum.

The very low concentration of organics in the emissions from Building 4L-5 (to the afterburner) in the first pilot test also resulted in the collection of an insufficient weight of sample in the VOST sampling system for characterization by gas chromatography/mass spectroscopy (GC/MS). To alleviate this problem in the second pilot test, Building 4L-5 emissions were measured using two much larger capacity Modified Method 5 (MM5) sampling trains over two separate sampling periods. The appropriate sampling time (and sampling temperatures) for these two sampling periods in the second pilot test were identified from the data collected during the first 26 hour pilot test run when the VOST sampling system was used.

7.2 Gas Sampling Equipment

During both pilot tests, the gas stream leaving the afterburner was sampled for a 4 to 5 hour period using an MM5 train while the inlet gas to the afterburner (gas stream leaving Building 4L-5) was also being sampled. In the first pilot test the VOST system was used to sample the hot gas leaving the west test area and entering the afterburner over the entire test period. In the second pilot test, two MM5 sampling trains sampled the hot gas leaving the east test area for two periods of about 5 hours each, one corresponding to maximum TNT emissions, and one corresponding to maximum hydrocarbon emissions.

For the safety reasons previously noted, it was necessary to remotely activate and deactivate both the VOST and MM5 sampling trains at the proper time from the control trailer located behind the embankment. Much greater reliability was required of these gas sampling systems than in the normal Environmental Protection Agency (EPA) sampling procedures because of the

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requirement for remote operation and the exclusion of personnel from the test area during the test (and sampling) period.

The objectives of our gas sampling program were twofold:

1. To the extent possible, define the quantity of and establish a time release profile for the release of organic material due to the thermal decontamination process; and

2. To verify that all the organic material liberated from the test area has been destroyed prior to its discharge to the environment.

To fulfill these program objectives, we designed and used three sampling systems employing three different sampling methodologies. Each of these systems was based on state-of-the-art technology.

The three gas sampling systems we used were:

*   a continuous total hydrocarbon analyzer;

*   a modified version of the EPA VOST system; and

*   an MM5 sampling system.

A summary description of each of these three systems is presented below.

More extensive descriptions, if so desired, of each of these sampling systems and methodologies can be found within the Test Plan[8] submitted to USATHAMA in May of 1986.

7.2.1 Total Hydrocarbon Analyzer

The volatile organic material (hydrocarbons) present in the heated air stream that exited the test area was monitored using a Beckman 402 Hydrocarbon Analyzer. Sample gas was delivered to the hydrocarbon analyzer through a heat traced line that was maintained at 240°F to prevent water condensation.

The Beckman 402 Hydrocarbon Analyzer is designed to measure the total hydrocarbon content of exhaust emissions. The analysis is based on flame ionization, a highly sensitive detection method. The instrument consists of:

*   a heated, temperature-controlled sample line;

*   an analyzer unit, incorporating a flame-ionization detector (FID) with associated critical sample-handling components contained within a temperature-controlled oven; and

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[8] Ibid Footnote 4.

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- an electronic unit, containing an electrometer amplifier and associated circuitry, readout meter, and recorder output provisions.

The hydrocarbon analyzer was used as a gross level detector for combustibles in the gas stream leaving the test structure. It provided real time monitoring that served three pur₁ᵣᵢses: (1) it cued the start of the VOST sampling; (2) provided an alarm if the hydrocarbon concentration reached 25% of the lower explosive limit (LEL); or (3) ultimately shutdown the system at a hydrocarbon concentration greater than 50% of LEL.

The hydrocarbon emission level in the first pilot test as measured by both the hydrocarbon analyzer (and the VOST sampling system) ranged from 10 to 150 ppm (vol/vol), well below the 22,000 ppm LEL for DMF and the other hydrocarbons liberated.

7.2.2 Modified VOST

Samples of the heated air exiting the test area, but prior to entering the afterburner, were collected, in the first pilot test using a sampling system that is derived from the EPA's VOST system. The principle differences between the system that we used and the standard EPA VOST system are: (1) a filter was inserted into the system upstream of the sorbent traps; (2) the second sorbent trap was packed only with activate charcoal; (3) the sorbents were chemically desorbed instead of thermally desorbed; and (4) instead of a single train, twelve separate trains were incorporated into the sampling system. (See Figure 7-1 for a schematic of this sampling system.)

In this VOST system, sample gas drawn from the duct was passed through a heated probe and a high efficiency quartz fiber filter. To avoid moisture condensation, the probe and filter assembly were maintained at a temperature of 120°C (250°F). Downstream of the filter assembly, the gas stream temperature was reduced to 70 to 90°F (somewhat higher than the 60 to 70°F optimum) in a water cooled heat exchanger prior to passing through a trap packed with approximately 2 grams of Tenax GC. A second sorbent trap, containing 3 to 5 grams of charcoal, was connected in series behind the Tenax sorbent trap. Condensed water percolated through both of these traps and was collected in a water knockout impinger located downstream of the charcoal trap. After the sorbent traps, the gas passed through a critical orifice, a pump, dry gas meter, and was then discharged.

To establish a time-release profile of organic material, twelve modified VOST sampling trains were connected in parallel in this system. Each of these remotely activated trains was used for a period of two to three hours (over the entire 26-hr test period) with a volume of roughly 20 to 30 liters of gas drawn through each train.

The final configuration of the VOST system employed in the first pilot test was different from the one presented in the Test Plan; there was both a change in the location of the solenoid valves and the addition of a water cooled heat exchanger. Changes in solenoid location were necessitated due to the fact that heat generated by the solenoid valves was being

7-3
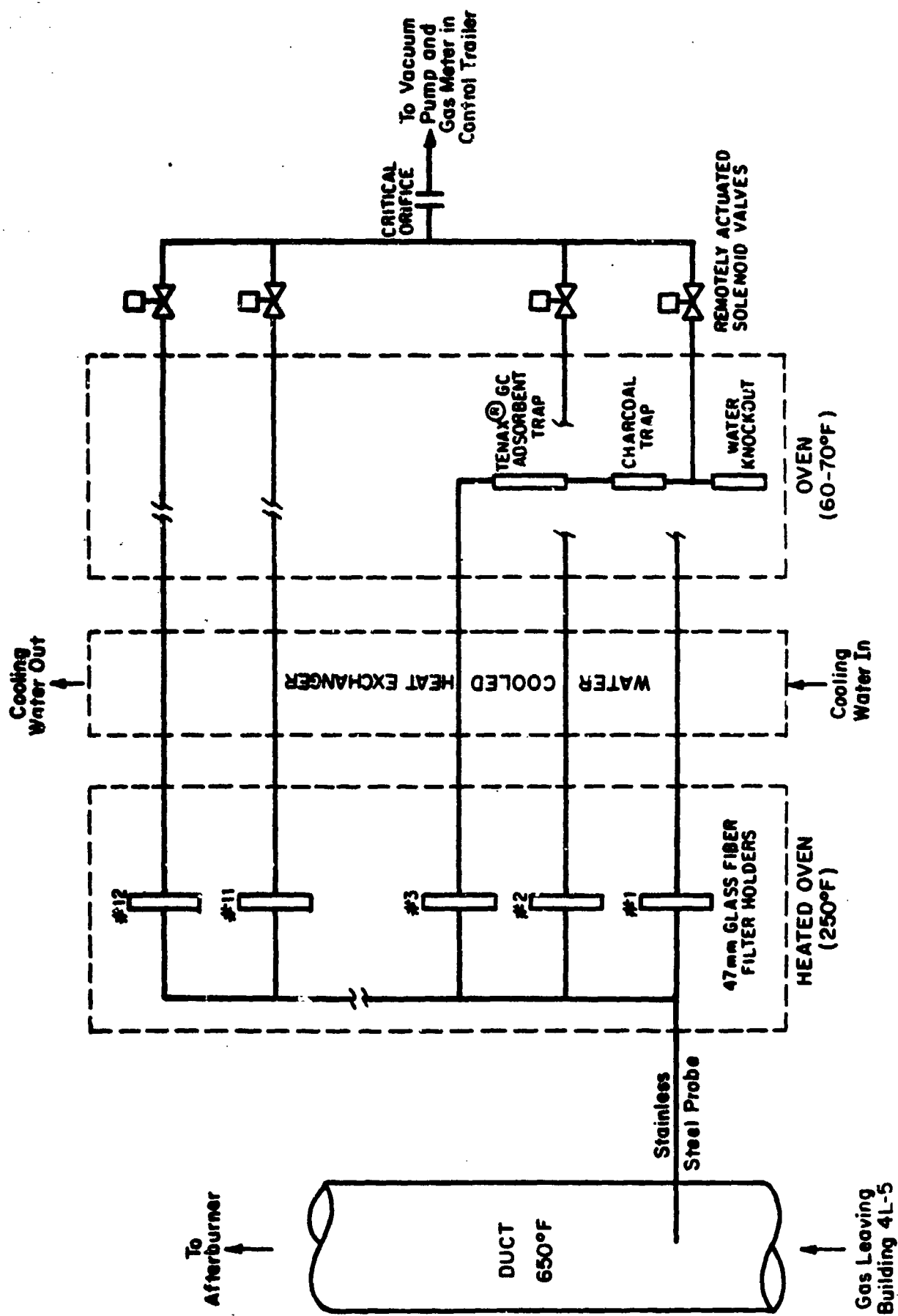
FIGURE 7-1 MODIFIED VOLATILE ORGANIC SAMPLING TRAIN (VOST)

Source: Arthur D. Little, Inc.

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transferred to the sampled gas stream if the solenoids were located upstream of the gas sampling system. The water cooled heat exchanger was added to the VOST system when the pilot test start date was changed from mid-winter to summer since the warmer ambient temperatures required cooling rather than heating of the sampled gases.

7.2.3 MM5 Sampling Train

The MM5 sampling system consisted of a stainless steel probe, a heated filter, a condenser, a sorbent module, and a series of five impingers. A diagram of the system configuration is given in Figure 7-2. In operation, a sample of exhaust gas was withdrawn from the stack at an isokinetic sampling rate into the probe of the sampling system. [An isokinetic sampling rate (i.e., sampling at a gas velocity through the probe equal to the velocity of the gas in the stack or duct) provides a representative sample of particulate from the sampled gas stream. If sampling is not done isokinetically, the differences in gas velocity at the tip of the probe will cause segregation of the different sizes of particulate]. The probe (in the stack) was cooled to slightly above 120°C (250°F) to ensure that water vapor did not condense before the sampled gas was passed through the high efficiency (99.95% efficient for 0.3 um DOP smoke particulate) quartz fiber filter. The filter itself was heated and maintained at 120°C (250°F) to minimize water condensation.

The sampled gas was then cooled to 21 to 32°C (70 to 90°F) within a water cooled condenser and drawn through a trap packed with XAD-2 adsorbent resin. This is the part of the sampling system where semi-volatile (e.g., boiling points 100 to 300°C) organic materials are concentrated. Moisture which condensed during this process was allowed to percolate through the adsorbent resin, and was collected in an empty impinger downstream of the sorbent trap. The sampled gas was then drawn through a series of four more impingers; two containing 100 mL each distilled water where moisture was condensed, one empty impinger, and then a fourth impinger containing silica gel where any traces of residual moisture were removed before the gas passed through a pump, a dry gas meter, and a flow limiting orifice.

A much more detailed description of the MM5 system and its operation is provided in the Arthur D. Little, Inc. Test Plan[9].

7.3 Gas Sampling Procedures

A number of changes had to be made in the gas sampling procedures as presented in the Test Plan in order to carry out the gas sampling for the pilot tests. A description of these revised gas sampling procedures is summarized below.

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[9] Ibid Footnote 4.

7-5
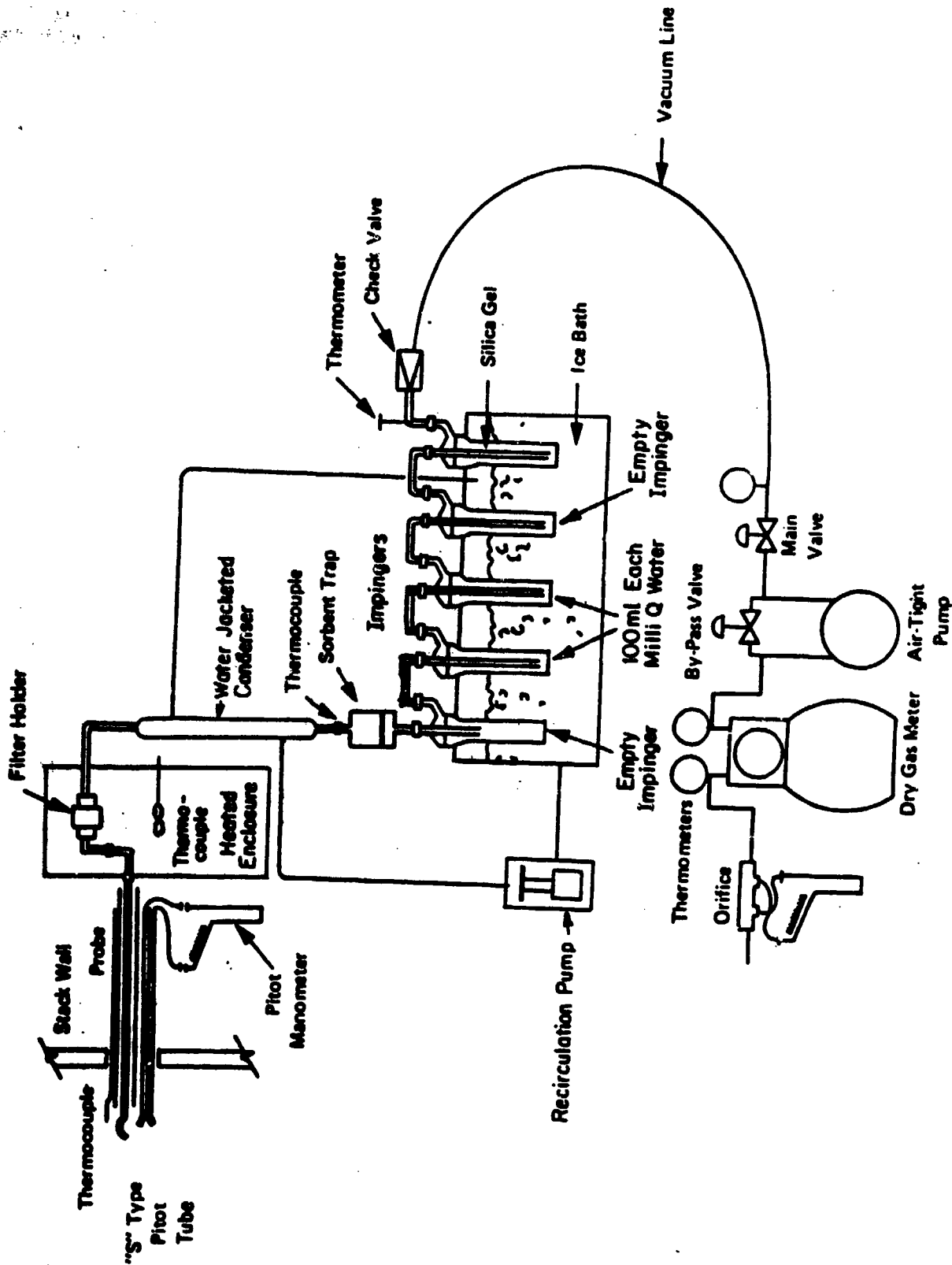
FIGURE 7-2 MODIFIED METHOD 5 (MM5) TRAIN SCHEMATIC DIAGRAM

Source: Arthur D. Little, Inc.

7.3.1 Total Hydrocarbon Analyzer

Acquisition of data with the total hydrocarbon analyzer proved to be very problematic. During initial set-up activities, the response on the analyzer was found to be very noisy. This problem was eventually corrected by replacing the flame ionization detector burner assembly, but subsequently the ability to properly hold span and zero on the analyzer was lost and a constantly drifting baseline was encountered.

Analytical results obtained from the total hydrocarbon analyzer were at best semi-quantitative. The highest concentration of organics measured by the hydrocarbon analyzer in the test area air outlet duct at any point throughout the test sequence was less than 100 ppm expressed as methane. Typically, this range drifted between 50 and 75 ppm of methane.*

Despite the operating problems, the hydrocarbon analyzer still measured hydrocarbon concentrations at about the same level as measured by the VOST sampling system.

7.3.2 Modified VOST

The only difficulty encountered during the VOST operation in the first pilot test (other than limited capacity which was discussed earlier in Section 7.1) was that it was not possible to control the temperature of the sorbent materials (Tenax GC and activated charcoal) at the desired level of 15 to 21°C (59 to 68°F). This difficulty arose due to the fact that the sorbent traps themselves were not temperature jacketed and the ambient temperatures encountered during the pilot testing ranged between 70 and 95°F. (The oven in which sorbent tubes were contained was originally designed for heating during winter operation.) Table 7-1 lists the sampling conditions for the 12 VOST trains.

7.3.3 MM5 Sampling Train

A sample of the emissions released from the afterburner in the first pilot test was collected using an MM5 sampling train. The entire sample volume was obtained from a point at the center of the afterburner exhaust duct at a sampling rate of roughly 0.167 standard cu m (0.6 scf @ 21°C and 760 mm Hg) per minute. Gas was withdrawn through a water cooled probe that was constructed entirely of stainless steel. Due to the fact that this sampling system was activated and operated from a distance of more than 500 ft, it was not possible to maintain the impinger bath at the prescribed temperature of 0 to 5°C (32 to 40°F). The bath temperature was maintained between 20 and 25°C (68 to 77°F), however, by continuously circulating cold water through the bath.

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*The total hydrocarbon analyzer used during this test sequence is a "carbon counter." Thus, a concentration of 1 ppm (vol/vol) of propane (C_3H_8) would give a response of 3 ppm (vol/vol) of methane (CH_4).

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TABLE 7-1

SUMMARY OF VOST SAMPLING

Sample No.	Start Time* Hrs	Date	Stop Time Hrs	Date	Avg. Dry Gas Meter Temp. (°F)	Volume at 29.92 in. Hg and 70°F (liters)
1	1400	6/18	1700	6/18	93.5	42.8
2	1700	6/18	2000	6/18	100.0	45.2
3	2000	6/18	2320	6/18	95.5	51.8
4	2320	6/18	0220	6/19	90.0	49.4
5	0220	6/19	0520	6/19	87.4	49.8
6	0520	6/19	0820	6/19	86.4	49.0
7	0820	6/19	1020	6/19	89.3	28.6
8	1020	6/19	1222	6/19	91.8	27.2
9	1222	6/19	1432	6/19	96.8	27.7
10	1432	6/19	1602	6/19	98.8	18.5
11	1602	6/19	1652	6/19	99.5	15.1
12	FIELD BLANK - NO SAMPLE TAKEN					

*Building air heater was started at 550°F at 1300 hrs on 6/18/86.

Source: Arthur D. Little, Inc.

At the beginning of the MM5 test, it was anticipated that a sample of roughly 6 cu m (210 cu ft) spanning a time period of 6 hrs would be obtained.

The actual duration of the MM5 test was 4.5 hours with roughly 4.5 cu m of gas sample obtained. The early shutdown was necessitated by the build-up of excessive pressure drop (27 in hg vacuum) through the system. This excessive pressure drop occurred rapidly between the 265 and 270 min period of the test, and was apparently caused by the partial fracture of the impinger containing silica gel. This fracture apparently allowed coolant bath water to contact the silica gel, forming a gel which plugged the tip of the impinger.

There were no problems with the three MM5 Trains used in the second pilot run even though it was necessary to remotely stop and restart the gas sampling during the second afterburner inlet sampling run.

The sample volumes collected, along with sampling conditions of the MM5 Train runs are presented in Table 7-2. The vapor content of the sampled gas stream was estimated for the first pilot run because it was unclear whether coolant bath water and impinger water had mixed when the silica gel impinger fractured.

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TABLE 7-2

SUMMARY OF MODIFIED METHOD FIVE (MM5) SAMPLING CONDITIONS

	First Pilot Test	Second Pilot Test			
	Afterburner Outlet	Afterburner Inlet 1	Afterburner Outlet 1	Afterburner Inlet 2
Air Heater Start Time	0013 hrs (6/18)	1445 hrs (10/15)	1445 hrs (10/15)	1445 hrs (10/15)
MM5 Start Time	0015 hrs (6/19)	1910 hrs (10/15)	1911 hrs (10/15)	0830 hrs (10/16)
MM5 Finish Time	0445 hrs (6/19)	0010 hrs (10/16)	0011 hrs (10/16)	1800 hrs (10/16)
Total Time (min)	270	300	300	310**
Dry Volume Sampled (cu ft)	155.0	214.0	183.5	218.3
Average Gas Meter Temperature (°F)	93.6	85.9	90.7	88.5
Average Barometric Pressure (in Hg)	29.96	30.30	30.27	30.25
Standard Dry Volume (cu ft) at 70°F & 29.92 in Hg	148.6	209.2	179.6	214.4
Volume Water Collected (mL)	255*	128.3	312.8	117.4
Volume of Water Vapor (cu ft) at 70°F 29.92 in Hg	12.0	6.0	14.7	5.5
Total Sample Volume at 70°F & 29.92 in Hg	160.6 cu ft (4.55 cu m)	215.2 cu ft (6.09 cu m)	194.3 cu ft (5.50 cu m)	219.9 cu ft (6.22 cu m)

*Sample may include some coolant bath water which was being supplied from a nearby fire hydrant.
**It was necessary to stop and restart this sampling run because of a minor fire that occurred during the testing.

Source: Arthur D. Little, Inc.

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/ℒ Arthur D. Little, Inc.

8.0 PILOT TEST RESULTS

8.1 Heat-Up Profile for Building 4L-5

8.1.1 West Test Area (Pilot Test 1)

The temperature distribution during heat-up of the inside of the west structural wall of Building 4L-5 (at 5, 10, 15 and 26 hrs) is shown in Figure 8-1. The heating throughout the test area was quite uniform with two exceptions. The low area of the south end of the west wall (thermocouples 4 and 9) and the area behind the hot air inlet duct (thermocouples 12, 14, 17, 18 and 38) heated up much more slowly than the rest of the wall due to poorer air circulation in those areas. The measured temperature differential between the hottest and coolest inner wall surface was, nevertheless, only about 78°F or about ± 40°F from the average. As expected, the upper wall areas heated more rapidly than the lower wall areas.

Figure 8-2 compares the maximum inside and outside wall surface temperatures of the west (structural) wall at the completion of the heat-up cycle. The temperature differential between the inside and outside of the wall as measured by the thermocouples was about 100°F for the areas of the wall subject to good air circulation. Those areas not subject to good air circulation (the north end and the lower south end of the west wall) had a greater temperature differential (140 to 200°F) across the wall and a lower final outside wall temperature (thermocouples 4 & 5 and 12 & 13). Heat-up time for the wall areas with poor air circulation was also greater, as expected. Figure 8-3 presents typical heat-up curves for the west wall (at thermocouple locations #1 and #2).

The inner surface of the contaminated blocks (thermocouples 19, 21, 31, and 33) in the center dividing wall reached maximum temperatures ranging from 620 to 645°F. The average surface temperature of these blocks (about 630°F) was nearly the same as the average temperature of the west wall at the same height directly across from them as read by thermocouples 8 and 14. Although the inside surface of the explosive contaminated blocks reached roughly the same temperature as the wall opposite (west wall of 4L-5), the outside surface of the contaminated blocks did not reach the same temperature as did the outside of the west wall. (Figure 8-4 shows the maximum interior/exterior temperatures of the contaminated test blocks.)

The initial heat-up rate of 50°F per hour of the walls for the first five hours was faster than the planned maximum of 30°F per hour because the minimum firing temperature of the air heater was 550°F rather than the 250°F originally expected. After the wall surface reached about 250°F, however, the air heater temperature was increased about 50°F per hour (until the air heater reached a temperature of 900°F) resulting in a wall surface temperature increase of about 20°F per hour. To bring the inside wall temperature up more rapidly to 600°F, the air heater temperature was increased to 1050°F at 24 hrs into the test run (after about 10 hrs at 900°F).

8-1
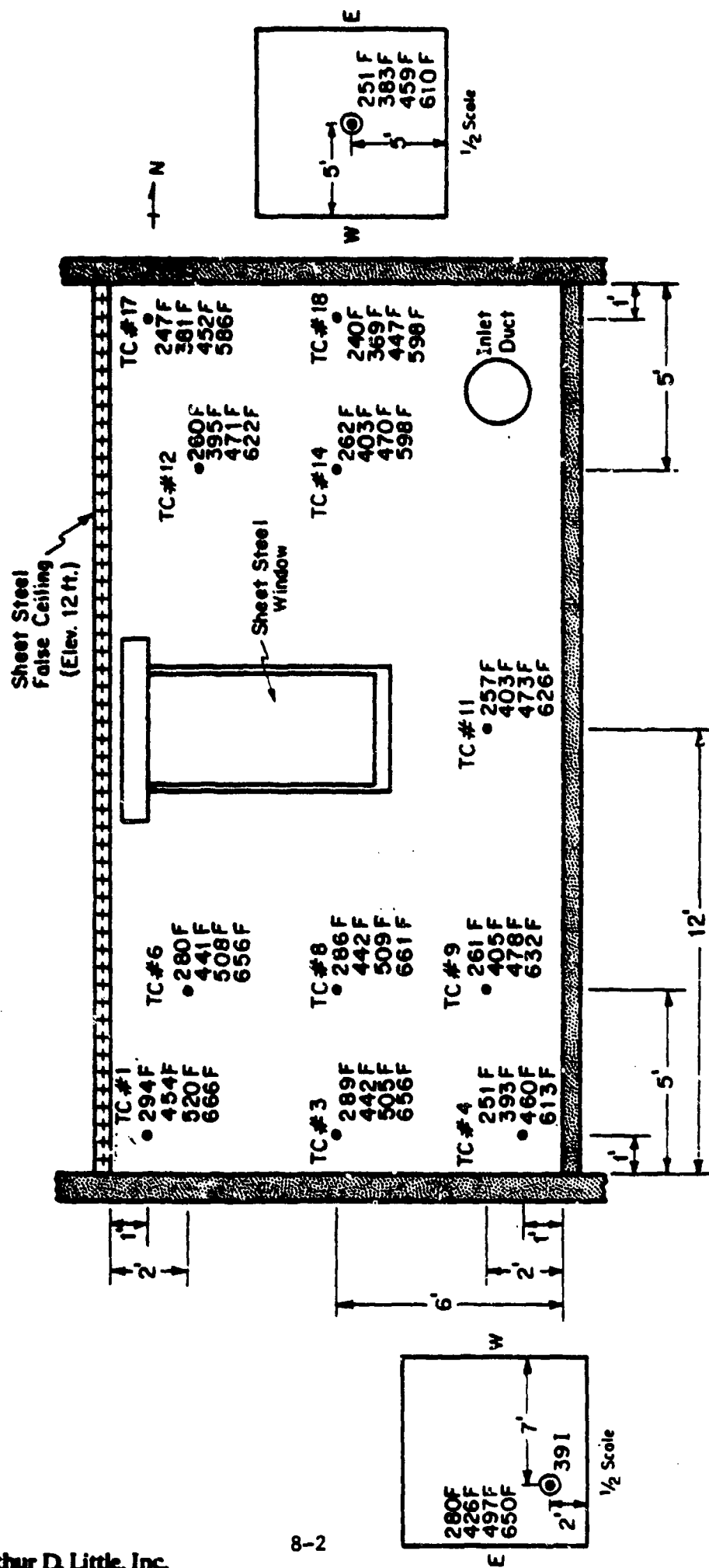
FIGURE 8-1 INSIDE SURFACE TEMPERATURES OF BUILDING 4L-5 WEST WALL AT 5, 10, 15, AND 26 HOURS – PILOT TEST 1

Source: Arthur D. Little, Inc.
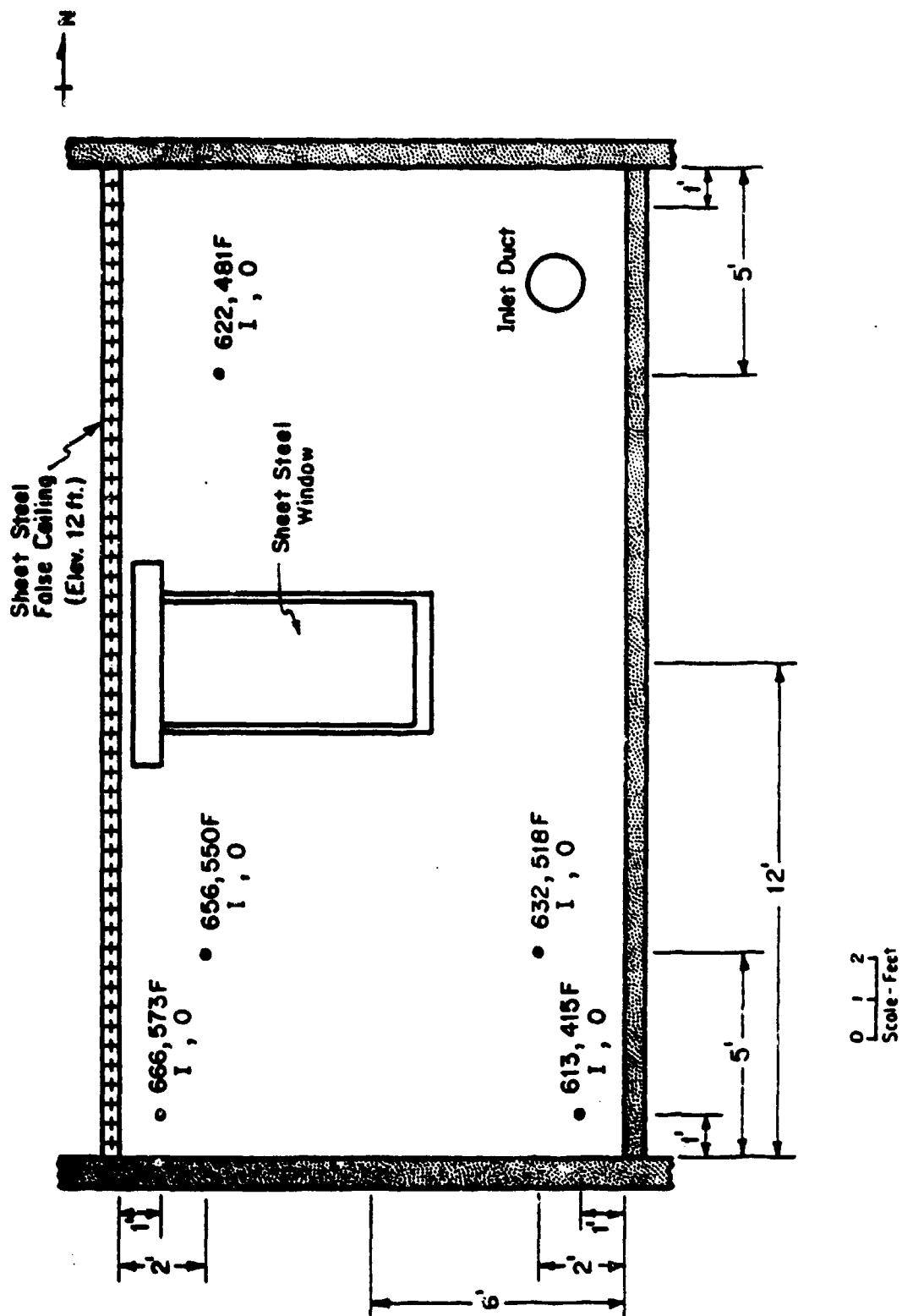
FIGURE 8-2 MAXIMUM INSIDE AND OUTSIDE SURFACE TEMPERATURES OF BUILDING
4L-6 WEST WALL (26 HRS) – PILOT TEST 1

Source: Arthur D. Little, Inc.
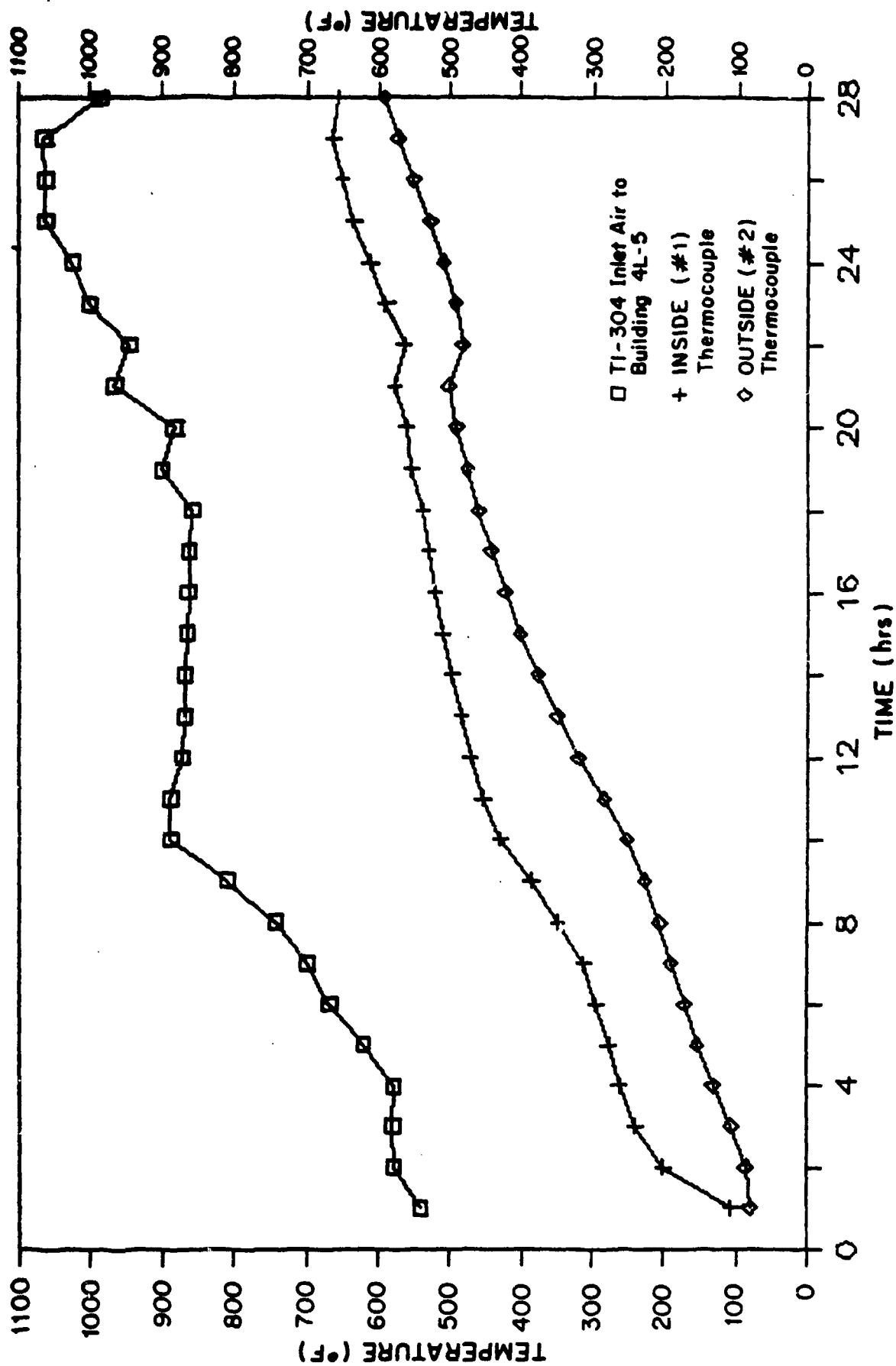
Arthur D. Little, Inc.

FIGURE 8-3 HEAT-UP CURVES FOR THERMOCOUPLES #1 AND #2 IN BUILDING 4L-5 WEST WALL – PILOT TEST 1

Source: *Arthur D. Little, Inc.*

Legend:
□ TI-304 Inlet Air to Building 4L-5
+ INSIDE (#1) Thermocouple
◇ OUTSIDE (#2) Thermocouple

8-4
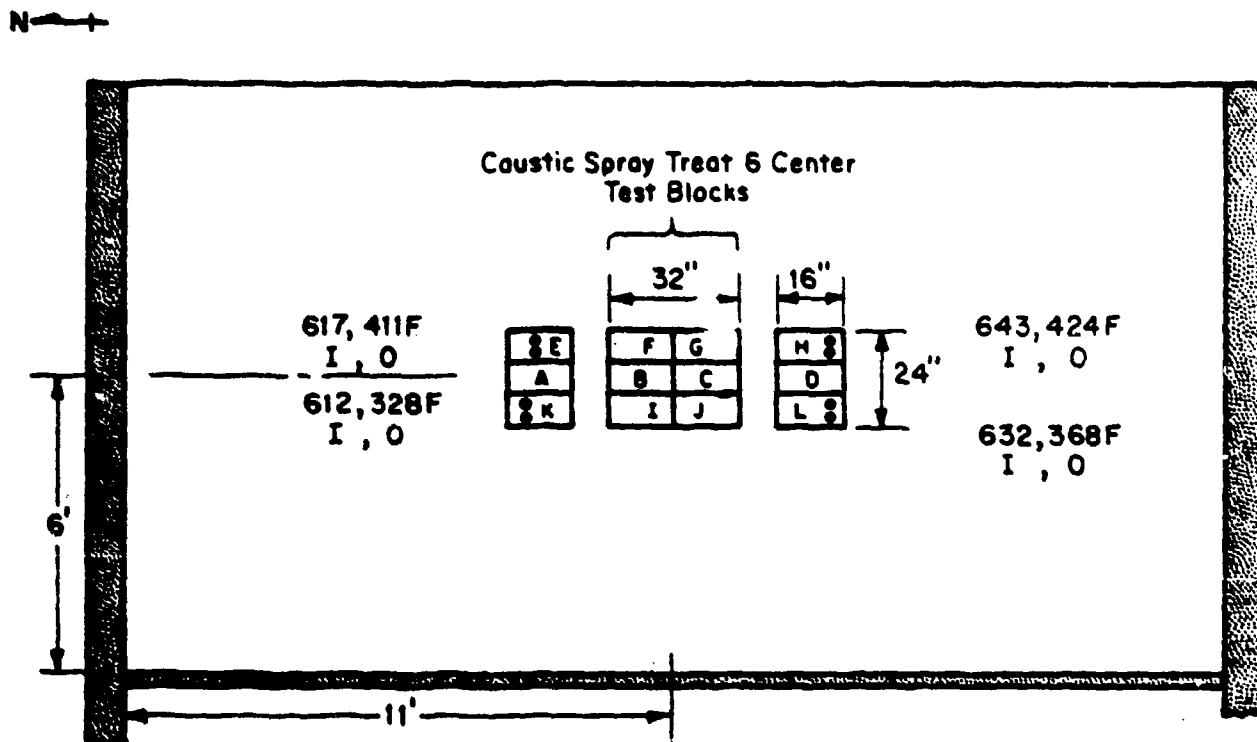
⚠ Arthur D. Little, Inc.

FIGURE 8-4 MAXIMUM INSIDE AND OUTSIDE SURFACE TEMPERATURES OF CONTAMINATED TEST CONCRETE BLOCKS IN BUILDING 4L-5 DIVIDING WALL (26 HRS) - PILOT TEST 1

Source: Arthur D. Little, Inc.

8.1.2 East Test Area (Pilot Test 2)

In the second pilot test, the air heater was started at a temperature of about 550°F and this inlet gas temperature to the test area held for about 4 hrs, the same initial conditions as used in the first pilot test. The air heater temperature was then increased at a rate of about 65°F per hour to 1000°F. At this point (about 11 hrs into the test run), a piece of insulation fell from the wall of the afterburner onto the lens of the UV flame detector causing a flameout indication and automatic shutdown of the entire burner system. When this fault was located and corrected (about one hour after shutdown or about 12 hrs into the pilot test run), the afterburner was restarted and brought up to temperature (1800°F) once again. The air heater was restarted at 550°F about 15 hrs into the second pilot test run, and brought up to 1050°F over a 3.5 hr period (a reheat rate of about 130°F per hour).

At 1025 hours on 10/16/86 (about 20 hrs into the test run) a small fire in the outside plywood insulation retaining wall was reported and the system was shut down again. The fire was quickly extinguished and the damaged plywood cut away. Approximately one hour later the afterburner was restarted at 850°F. The air heater was restarted about two hours after shut down (at 550°F) when the afterburner temperature had reached about 1400°F. (As the air heater temperature reached 1000°F, the afterburner temperature had reached its final operating temperature of 1800°F.)

The air heater was brought back up to 1050°F by 1445 hours and held at this temperature until the completion of testing at 1830 hours on 10/16/86.

A plot of the east inside wall temperature (at two points) and the inlet gas duct temperature is given in Figure 8-3.

As can be seen by the temperature plots, the east test area was subjected to a much more severe temperature cycling in this second pilot test than was the west test area in the first pilot test. This was further evidenced by the fact that while the west test area showed no new cracks in the walls or floor or widening of existing cracks after heating, the east test area showed both some new cracks in the wall and some widening of the existing cracks in the wall and floor.

8.2 Heat Transfer Rate

Table 8-1 summarizes the data pertaining to heat transfer to the west test area during the first pilot run. Table 8-2 presents sample heat transfer calculations. Based on the data presented in Table 8-1, the overall heat transfer coefficients we have calculated for heat transfer to the test area surfaces are as follows:

    Walls   -   2.0 Btu/hr sq ft °F
    Floor   -   2.4 Btu/hr sq ft °F
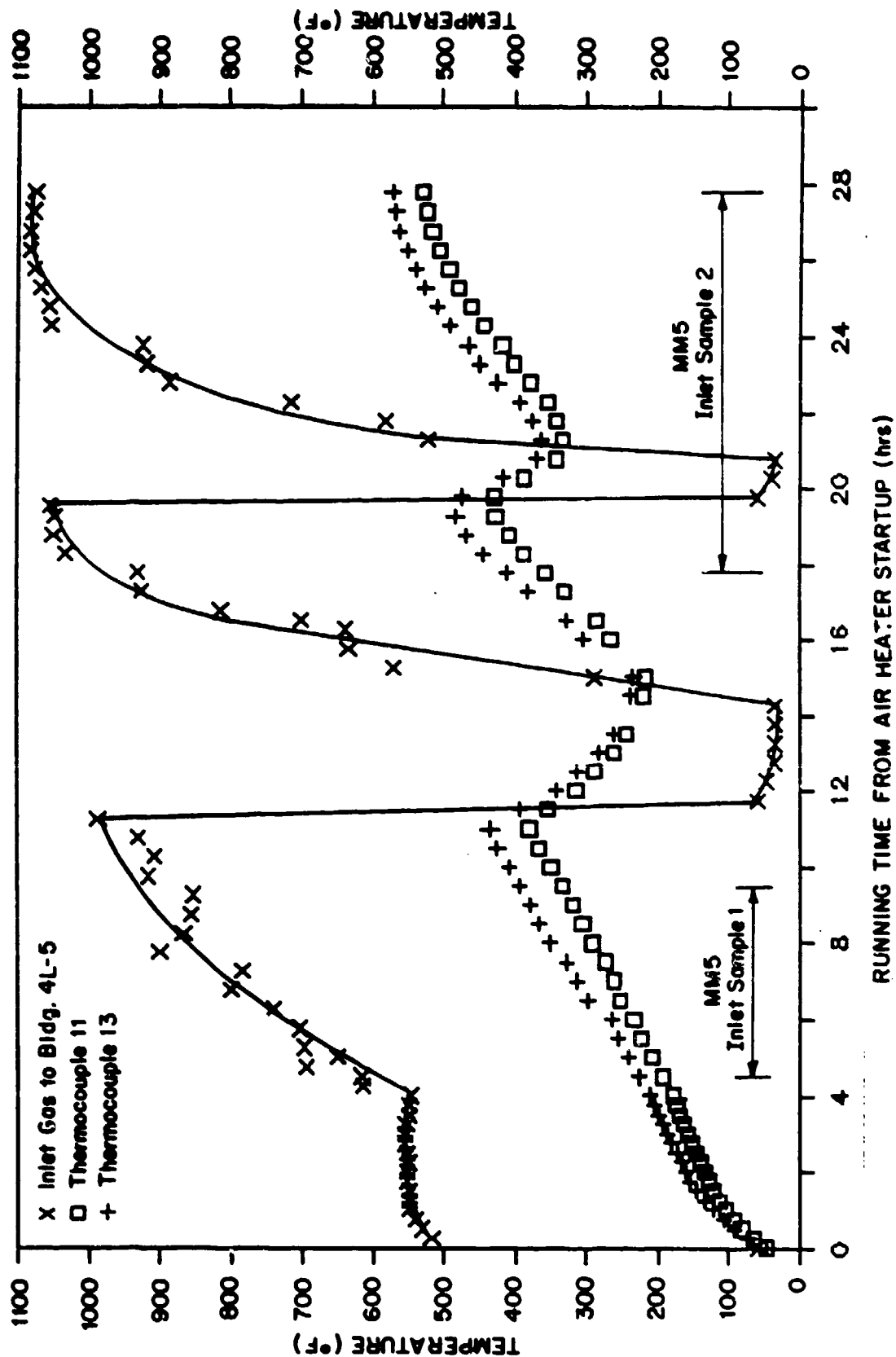
Arthur D. Little, Inc.

FIGURE 8-5 INLET GAS AND SELECTED WALL SURFACE TEMPERATURES – PILOT TEST 2

Source: Arthur D. Little, Inc.

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TABLE 8-1

SUMMARY OF HEAT TRANSFER DATA - PILOT TEST 1

Running Time (hours)	Temp. Out (F)	Temp. In (F)	Net Heat Input (mmBTU/hr)	Heat Absorbed Wall --(mm	Floor /hr)---	Losses Roof	Wall (mmBTU/HR)	Floor	Unacctd
0.00	80	290	0.41	0.43	1.10	0.01	0.01	0.03	-1.17
0.25									
0.50	273	571	0.58	0.36	0.34	0.02	0.04	0.06	-0.25
0.75									
1.00	301	578	0.53	0.29	0.17	0.02	0.05	0.07	-0.07
1.25									
1.50	311	579	0.51	0.25	0.13	0.02	0.06	0.08	-0.03
1.75									
2.00	319	579	0.49	0.23	0.12	0.02	0.06	0.08	-0.02
2.25									
2.50	325	579	0.48	0.21	0.11	0.02	0.07	0.08	-0.01
2.75									
3.00	327	578	0.47	0.18	0.10	0.02	0.07	0.08	0.02
3.25									
3.50	332	582	0.47	0.17	0.09	0.02	0.06	0.08	0.05
3.75									
4.00	338	619	0.53	0.16	-0.06	0.02	0.06	0.08	0.27
4.25									
4.50	348	668	0.61	0.17	0.08	0.02	0.06	0.08	0.20
4.75									
5.00	355	667	0.59	0.18	0.14	0.02	0.05	0.08	0.12
5.25									
5.50	365	699	0.63	0.17	0.08	0.02	0.07	0.08	0.22
5.75									
6.00	368	698	0.63	0.15	0.22	0.02	0.07	0.08	0.09
6.25									
6.50	391	736	0.66	0.26	0.36	0.02	0.07	0.10	-0.14
6.75									
7.00	404	761	0.65	0.23	0.22	0.02	0.07	0.10	0.00
7.25									
7.50	430	813	0.74	0.25	0.18	0.03	0.08	0.11	0.10
7.75									
8.00	437	808	0.71	0.29	0.30	0.03	0.09	0.11	-0.11
8.25									
8.50	447	892	0.84	0.25	0.17	0.03	0.09	0.12	0.18
8.75									
9.00	474	887	0.79	0.24	0.37	0.03	0.10	0.12	-0.06
9.25									
9.50	482	887	0.77	0.19	0.12	0.03	0.10	0.11	0.22
9.75									
10.00	489	887	0.76	0.23	0.11	0.03	0.10	0.11	0.18
10.25									
10.50	493	895	0.77	0.18	0.00	0.03	0.10	0.12	0.34
10.75									
11.00	495	871	0.71	0.17	0.03	0.03	0.10	0.12	0.27
11.25									
11.50	499	870	0.70	0.17	0.04	0.03	0.09	0.12	0.25
11.75									
12.00	503	869	0.69	0.17	0.03	0.03	0.09	0.12	0.25
12.25									
12.50	506	866	0.66	0.16	0.03	0.03	0.09	0.12	0.24
12.75									
13.00	510	868	0.67	0.16	0.03	0.03	0.09	0.12	0.24
13.25									
13.50	512	867	0.66	0.16	0.03	0.03	0.08	0.12	0.24
13.75									
14.00	515	866	0.66	0.15	0.02	0.03	0.08	0.13	0.25
14.25									
14.50	517	865	0.65	0.15	0.03	0.03	0.08	0.13	0.24

Note: Temp in at 1850 SCFM, Temp out at 2000 SCFM.

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TABLE 8-1 (continued)

SUMMARY OF HEAT TRANSFER DATA - PILOT TEST 1

Running Time (hours)	Temp. Out (F)	Temp. In (F)	Net Heat Input (mmBTU/hr)	Heat Absorbed Walls (mmBTU/hr)	Heat Absorbed Floor (mmBTU/hr)	Losses Roof (mmBTU/HR)	Losses Wall (mmBTU/HR)	Losses Floor (mmBTU/HR)	Losses Unacctd (mmBTU/HR)
14.75									
15.00	519	864	0.64	0.13	0.02	0.03	0.08	0.13	0.25
15.25									
15.50	523	863	0.63	0.13	0.02	0.03	0.08	0.13	0.24
15.75									
16.00	526	863	0.62	0.13	0.02	0.03	0.07	0.13	0.24
16.25									
16.50	529	860	0.61	0.12	0.01	0.03	0.07	0.13	0.25
16.75									
17.00	531	859	0.61	0.12	0.02	0.03	0.07	0.13	0.23
17.25									
17.50	538	885	0.64	0.15	0.09	0.03	0.06	0.14	0.18
17.75									
18.00	544	899	0.67	0.14	0.07	0.03	0.07	0.14	0.23
18.25									
18.50	545	889	0.64	0.11	0.06	0.03	0.07	0.14	0.23
18.75									
19.00	545	884	0.63	0.10	0.05	0.03	0.07	0.14	0.25
19.25									
19.50	545	890	0.65	0.09	0.05	0.03	0.07	0.14	0.28
19.75									
20.00	560	966	0.78	-0.36	-1.01	0.03	0.07	0.15	1.90
20.25									
20.50	Air Heater Down		0.00	0.35	1.04	0.00	0.06	0.13	-1.57
20.75									
21.00	553	947	0.76	0.15	0.13	0.03	0.06	0.14	0.24
21.25									
21.50	568	974	0.78	0.20	0.21	0.03	0.07	0.15	0.13
21.75									
22.00	582	1000	0.80	0.15	0.08	0.03	0.07	0.15	0.31
22.25									
22.50	588	999	0.79	0.17	0.11	0.03	0.07	0.15	0.25
22.75									
23.00	598	1026	0.82	0.16	0.10	0.03	0.08	0.15	0.30
23.25									
23.50	610	1057	0.86	0.19	0.12	0.03	0.08	0.16	0.28
23.75									
24.00	617	1062	0.85	0.18	0.06	0.04	0.08	0.16	0.34
24.25									
24.50	622	1062	0.84	0.16	0.06	0.04	0.08	0.16	0.35
24.75									
25.00	627	1062	0.83	0.15	0.05	0.04	0.08	0.16	0.35
25.25									
25.50	632	1065	0.83	0.14	0.07	0.04	0.08	0.16	0.34
25.75									
26.00	635	1065	0.82	0.15	0.05	0.04	0.08	0.17	0.34
26.25									
26.50	626	997	0.69	0.02	-0.15	0.03	0.07	0.16	0.55
26.75									
27.00	619	986	0.69	0.06	0.00	0.03	0.06	0.16	0.38

Note: Temp in at 1850 SCFM, Temp out at 2000 SCFM.

Source: Arthur D. Little, Inc.

TABLE 8-2

SAMPLE HEAT TRANSFER CALCULATIONS

Sample calculations are based on data for Pilot Run 1, Hour 11.00.

Air temperature into test area:

T_{in} = 871°F (by thermocouple)

Air temperature out of test area:

T_{out} = 495°F (by thermocouple)

Net Heat Input:

$$H = (\dot{m})(t)(\rho)(Cp)[(T_{in} - T_{amb}) - 1.081(T_{out} - T_{amb})]$$

Where: Air flow into room ($\dot{m}$) = 1850 scfm   (from dp cell)

Air infiltration = 150 scfm (estimated at 8% of air flow in)

Air flow leaving room = 2000 scfm

Air density (ρ) = 0.0733 lb/scf

Cp_{air} = 0.26 Btu/lb°F

$T_{ambient}$ = 60°F

Time period(t) = 60 min

$$H = (1850)(60)(.0733)(0.26)[(871-60) - 1.081(495-60)]$$
$$= 0.721 \times 10^6 \text{ Btu/hr}$$

Heat Absorbed-Walls:

$$H_{walls} = (M)(Cp)(\Delta T)(2)$$

Where: Weight per block = 37.7 lbs

Total weight of walls (M) = 37,660 lbs

Cp_{walls} = 0.25 Btu/lb°F

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TABLE 8-2 (continued)

SAMPLE HEAT TRANSFER CALCULATIONS

ΔT = Average wall temperature change over 1/2 hour

($\pm$ 15 mins. from run hour 11.00)

@ Run Hour 10.75, T_{wall} = 348.5°F (by thermocouple)

@ Run Hour 11.25, T_{wall} = 357.5°F (by thermocouple)

ΔT = 9.0°F

H_{walls} = (37,660)(0.25)(9.0)(2)

= 0.17 x 10^6 Btu/hr

Heat Absorbed-Floor:

H_{floor} = (M)(Cp)(ΔT)(2)

Where: Density of floor = 137 lb/ft^3

M_{floor} = 27,390 lbs

Cp_{floor} = 0.27 Btu/lb F

ΔT = Average floor temperature change over 1/2 hour

($\pm$ 15 mins. from run hour 11.00)

@ 10.75 T_{floor} = 377.6°F (by thermocouple)

@ 11.25 T_{floor} = 379.5°F (by thermocouple)

ΔT = 1.9°F

H_{floor} = 27,390 (0.27)(1.9)(2)

= 0.028 x 10^6 Btu/hr

Heat Losses-False Ceiling:

$Q_{loss \cdot ceiling}$ = (U)(A)(ΔT)

Where: U = overall heat transfer coefficient (ceiling to outside bldg)

= 0.15 Btu/hr ft^2·F (estimate)

A = ceiling area

= 300 ft^2

ΔT = (T_{bulk} - $T_{ambient}$) where T_{bulk} is avg. of T_{in} and T_{out}

T_{amb} = 60°F

T_{bulk} = (871 + 495)/2 = 683°F

TABLE 8-2 (continued)

SAMPLE HEAT TRANSFER CALCULATIONS

$$Q_{loss-ceiling} = (0.15)(300)(683 - 60)$$
$$= 0.028 \times 10^6 \text{ Btu/hr}$$

Heat Losses-Wall:

$$Q_{loss-wall} = A \left(\frac{k}{x}\right) \Delta T$$

Where: A ~ wall area = 888 sq ft

      k = thermal conductivity of wall

        = 0.439 Btu/hr sq ft °F per ft

      x = wall thickness

        = 0.667 ft

    ΔT = average wall temperature gradient = $(T_{inside} - T_{outside})$

        T_{inside} = 435.5°F (by thermocouple)

        $T_{outside}$ = 270.5°F (by thermocouple)

$$Q_{loss-wall} = 888 \ (.439/.667)(435.5 - 270.5)$$
$$= 0.096 \times 10^6 \text{ Btu/hr}$$

Heat Losses-Floor:

$$Q_{loss-floor} = A \left(\frac{k}{x}\right) \Delta T$$

Where: A = 300 sq ft

      k = 0.792 Btu/hr sq ft °F per ft

      x = 0.667 ft

    ΔT = average floor temperature gradient = $(T_{top} - T_{bottom})$

        T_{top} = 541.8°F (by thermocouple)

        T_{bottom} = 214.3°F (by thermocouple)

$$Q_{loss-floor} = 300 \ (0.792/0.667)(541.8 - 214.3)$$
$$= 0.117 \times 10^6 \text{ Btu/hr}$$

TABLE 8-2 (continued)

SAMPLE HEAT TRANSFER CALCULATIONS

Heat Losses-Unaccounted:

$Q_{unaccounted}$ = [(net heat input) - (heat absorbed walls)

- (heat absorbed floors) - (heat loss ceiling)

- (heat loss walls) - (heat loss floor)] x 10^6

= (0.71 - 0.17 - 0.03 - 0.03 - 0.10 - 0.12) x 10^6

= 0.27 x 10^6 Btu/hr

Heat Transfer Coefficient (Gas to building inside surface):

$h_{gas\ to\ wall}$ = $(Q_{to\ wall})/(A \Delta T)$

Where:

$$Q_{unacct\ walls} = \frac{Q_{abs} + Q_{loss}\ (walls)}{Q_{abs} + Q_{loss}\ (walls + floor + ceiling)} \times Total\ Q_{unacct}$$

$$= \frac{0.17 + 0.10}{0.17 + 0.03 + 0.03 + 0.10 + 0.12} \times 0.27 \times 10^6$$

$$= 0.16 \times 10^6$$

$Q_{to\ wall}$ = $Q_{abs} + Q_{loss} + Q_{unacct}$ = (0.17 + 0.10 + 0.16) x 10^6

= 0.43 x 10^6 Btu/hr

A = 888 sq ft

$\Delta T = (\frac{T_{in} + T_{out}}{2}) - T_{wall} = \frac{(871+495)}{2} - 435.5 = 247.5$ °F

$h_{gas\ to\ wall}$ = 0.43 x 10^6/(888)(247.5)

= 2.0 Btu/hr sq ft °F

Note: Some data has been rounded.

Source: Arthur D. Little, Inc.

▲ Arthur D. Little, Inc.

The heat transfer coefficient calculated below using a modification of the equation reported by V. Ganapathy[10] predicts a somewhat higher value than was actually measured in the pilot tests.

$$Q = 0.296 (T_g - T_s)^{1.25} + 0.174e [(T_g/100)^4 - (T_s/100)^4]$$

where: Q = Btu/hr sq ft

T_g = gas temperature (average), °R

T_s = surface (wall) temperature, °R

e = effective emittance

U = overall heat transfer coefficient (Btu/hr sq ft °F)

And for the walls where:

T_s = 460 + 436 = 896°R

$$T_g = 460 + \frac{871 + 495}{2} = 1143°R$$

$e_{concrete}$ = 0.63

e_{gas} = 0.40

$$e_{effective} = \frac{1}{\frac{1}{0.63} + \frac{1}{0.40} - 1} = 0.32$$

$$Q_{wall} = 0.296(1143-896)^{1.25} + 0.174(.32) [11.43^4 - 8.96^4]$$

$$= 290 + (0.174)(0.32)(10623) = 880 \text{ Btu/hr sq ft}$$

$$h_{to\ wall} = \frac{Q_{wall}}{A \Delta T} = \frac{880 \text{ Btu/hr sq ft}}{(1)(683-436)°F} = 3.6 \text{ Btu/hr sq ft °F}$$

The overall heat transfer coefficients actually determined in Pilot Test 1 were used (in Section 10.0 of this report) for estimating the size and cost of a burner system to treat two different size building areas with dimensions of 100 ft x 60 ft x 15 ft ht and 40 ft x 30 ft x 12 ft ht.

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[10]V. Ganapathy, "Relating Heat Emission to Surface Temperature," Calculation and Shortcut Deskbook, McGraw-Hill, Inc., p. 20.

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8.3 Effect of Heating on Building 4L-5 Structural Integrity

A number of techniques were used for evaluating the effect of heating on the structural integrity of two types of materials of construction; concrete block and concrete. These included metrology, bend strength tests, compressive strength tests, and X-ray diffraction analysis. Each of these techniques is discussed in more detail below.

8.3.1 Metrology

Horizontal and vertical lines (forming a 12 ft x 24 ft grid) were painted on the west wall of Building 4L-5 (with the aid of a level) using heat resistant (1000°F) paint in Pilot Test 1. This heat resistant paint was applied over the existing paint on the wall. After the test, during which the painted wall surface (west wall) was heated to over 600°F, it was discovered that most of the non-heat resistant paint had been burned off the wall surface taking most of the lines painted in heat resistant paint with it. Enough of the heat resistant paint remained on the wall, however, to allow chalk lines to be snapped to reconstruct the grid and to confirm there had been no major shifting (or discontinuity) of the walls caused by the heating and cooling of the building.

In addition to the fact that there were no breaks or distortion of the grid lines painted on the wall, there were no indications of any new cracks in the wall, widening of the existing cracks, or any other visible damage to the building as a result of the heating cycle.

8.3.2 Bend Strength Tests

Bend strength tests rather than compressive strength tests were conducted o core samples cut from the structural (west and east) concrete block walls of Building 4L-5 after the two pilot tests. This type of test is more sensitive to the microscopic fracture lines induced by thermal stress that will ultimately cause compressive failure and much less costly to perform than the compressive strength test. There also tends to be a good correlation between compressive strength (compressive load only) and bend strength (a combination of compressive and tensile loads). Compressive strength is usually about five to eight times the bend strength for concrete.[11]

All of the bend tests were performed in a type TT Instron testing machine using a crosshead speed of 1.0 cm per minute. A three-point fixture having a span of 6.30 cm was used in conjunction with the Instron to apply the load to each specimen. The bars were cut to a nominal nine cm length and a square cross section (width and depth) of about two cm.

The test bars were cut from each of the concrete blocks by diamond sawing. Initially, a rotary-masonary saw was used to cut the specimens to length

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[11]O'Rourke, et al., "General Engineering Handbook," (2nd Edition), McGraw-Hill Book Co., Inc. (1940).

and to "rough cut" the width and depth. A wafering machine, again a rotary-type, was used to provide flatness and parallelism to the specimens. For both of these saws the feed is manual. Subsequent to sawing, the edges of the specimens were rounded by use of a silicon carbide belt sander. For each of these sawing and lapping operations, the specimens were flooded with water to minimize heat build-up, therefore, it was necessary to dry each specimen in a recirculating air oven at about 50°C for a period of 18 hrs (overnight) before testing.

Figure 8-6 is a plot of concrete block specimen bend strength (in psi) versus maximum structural wall concrete block temperature. About two-thirds of the curve is presented as a dotted line, since the shape of this portion of the curve is not well defined due to the scatter of the data. It is evident from the data, however, that there is about a 70% decrease in bend strength in going from unheated concrete blocks to concrete blocks heated up to a maximum of 700°F. Concrete blocks heated to only 400°F exhibit a loss in bend strength of about 45%. (Appendix C provides a detailed summary of bend test results.)

8.3.3 Comparison of Bend and Compressive Strength for Heated Block Samples

In an attempt to better define the relationship between loss in bend strength versus loss in compressive strength after heating, specimens for both bend and compressive strength tests were cut from a new concrete block. The specimens, to be used for both bend and compressive strength tests, were then heated together in a laboratory oven to different temperatures. The heating cycle for the samples was as follows:

    400°F Specimens (5 hours @ 400°F)
    500°F Specimens (1 hour @ 400°F, 3 hours @ 500°F)
    600°F Specimens (1 hour @ 400°F, 1 hour @ 500°F, 2 hours @ 600°F)

The compressive strength samples were cut to approximately 3 cm* by 3 cm square cross section by 7 cm in length prior to heat treatment.* To aid in alignment and to reduce bending moments in the specimens during loading, a sulfur mortar (as described in ASTM Standard C617) was used for end caps after heating the specimens. This material was cast to about 0.5 cm thickness on both ends of each test specimen.

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*According to Mr. Ara Shrestinian of the Thompson & Lichtner Company (Consulting Engineers - Engineering & Testing Laboratories, Cambridge, MA), there is no ASTM standard for compressive strength of concrete blocks. Usually they will test a whole concrete block in their compression tests. For cast concrete, a 4 inch diameter cylinder is specified by ASTM C-42. For hydraulic cement (no aggregate) ASTM Standard C-109 specifies 2 in. or 50 mm cubes for compressive strength testing. We attempted to obtain cylindrical specimens from the concrete blocks for testing, but all the cylindrical samples fractured in the coring process.
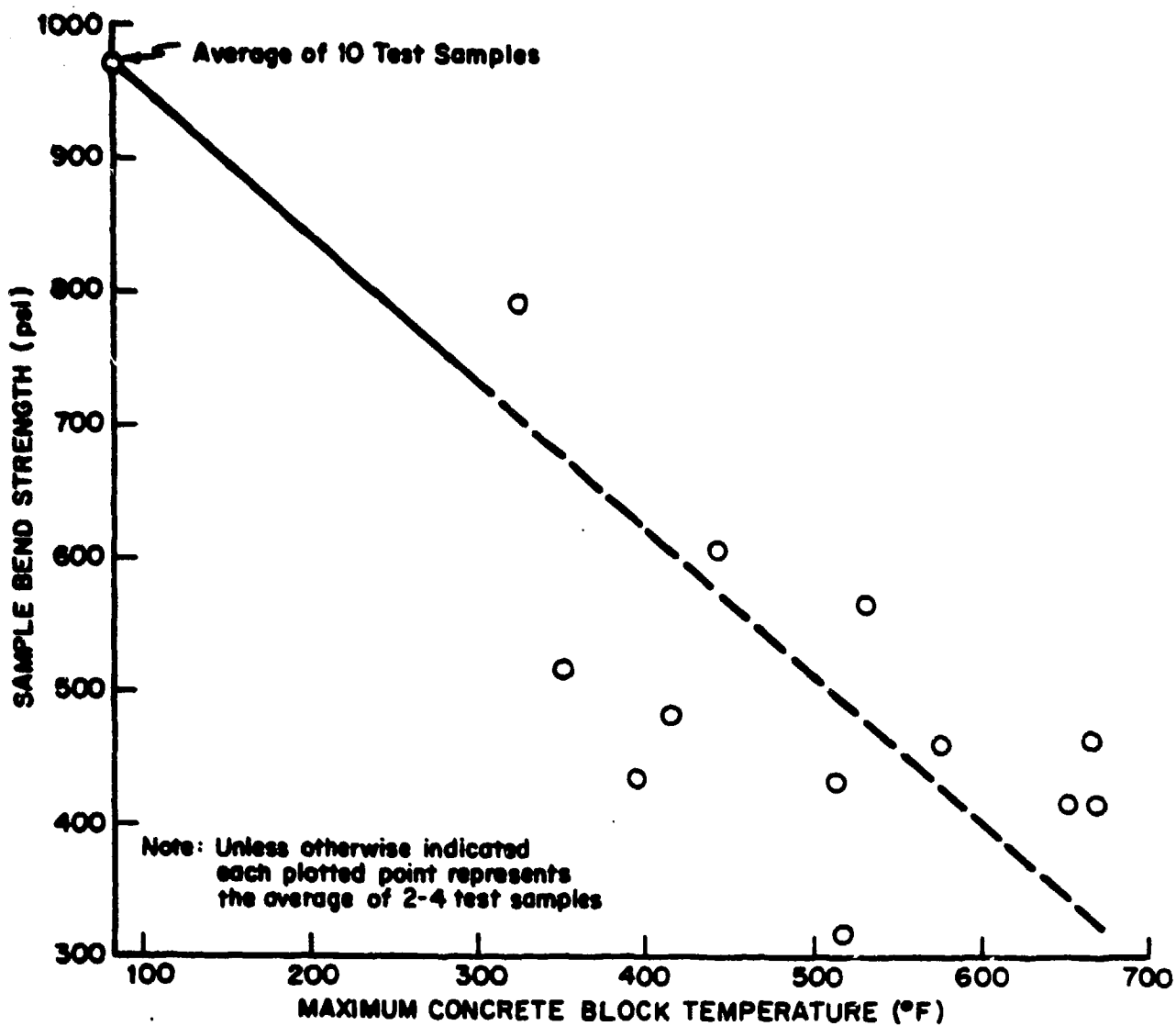
FIGURE 8-6 EFFECT OF MAXIMUM TEMPERATURE ON BEND
STRENGTH OF BUILDING 4L-5 STRUCTURAL WALL
BLOCK SAMPLES - PILOT TESTS 1 and 2

Source: Arthur D. Little, Inc.

Arthur D. Little, Inc.

The load was applied to each specimen by an Instron testing machine. A constraint cross-head speed of 1 mm per minute was used to load the specimens to failure. Prior to testing, dimensions for each specimen were obtained by micrometer measurement. These measurements were then used to calculate the cross sectional area of each specimen. The load was measured and noted at the point of failure for each specimen and the resultant compressive strength was calculated as:

$$S_c \text{ [compressive strength (psi)]} = \frac{\text{load at failure/specimen cross section}}{\text{(lbs)} \quad [d(in) \times b(in)]}$$

The samples for bend tests were prepared in the same manner (prior to heating) as described in Section 8.3.2, except the test bars were cut to a size of 2.5 cm x 2.5 cm x 9 cm long. The bend and compressive strengths determined for the heated (and unheated) test samples are plotted in Figure 8-7. (Appendix C provides a detailed summary of compressive strength results.)

The ratios between the bend strength and compressive strength for these samples were as follows:

New Concrete Block Sample	Average Bend Strength (psi)	Average Compressive Strength (psi)	Ratio of Compressive Strength to Bend Strength
Unheated	621	3400	5.5
400°F	521	3215	6.2
500°F	388	3069	7.9
600°F	398	2895	7.4

Although the ratio between the compressive and bend strengths stayed within the 5 to 8 range, the loss in tensile strength (and, therefore, bend strength) apparently was much greater (as shown in Figure 8-7 and in the data summary below) than the loss in compressive strength.

New Concrete Block Sample	Percent of Bend Strength of Unheated Sample	Percent of Compressive Strength of Unheated Sample
Unheated	100.0	100.0
400°F	83.9	94.6
500°F	62.4	90.3
600°F	64.1	85.1

Samples were also taken from the concrete floor and concrete lintels over the windows in the east test area for compressive strength testing before and after the second pilot test. As shown below, the loss in compressive strength with heating for these concrete samples appeared to be much greater than the loss in compressive strength of the new concrete block.

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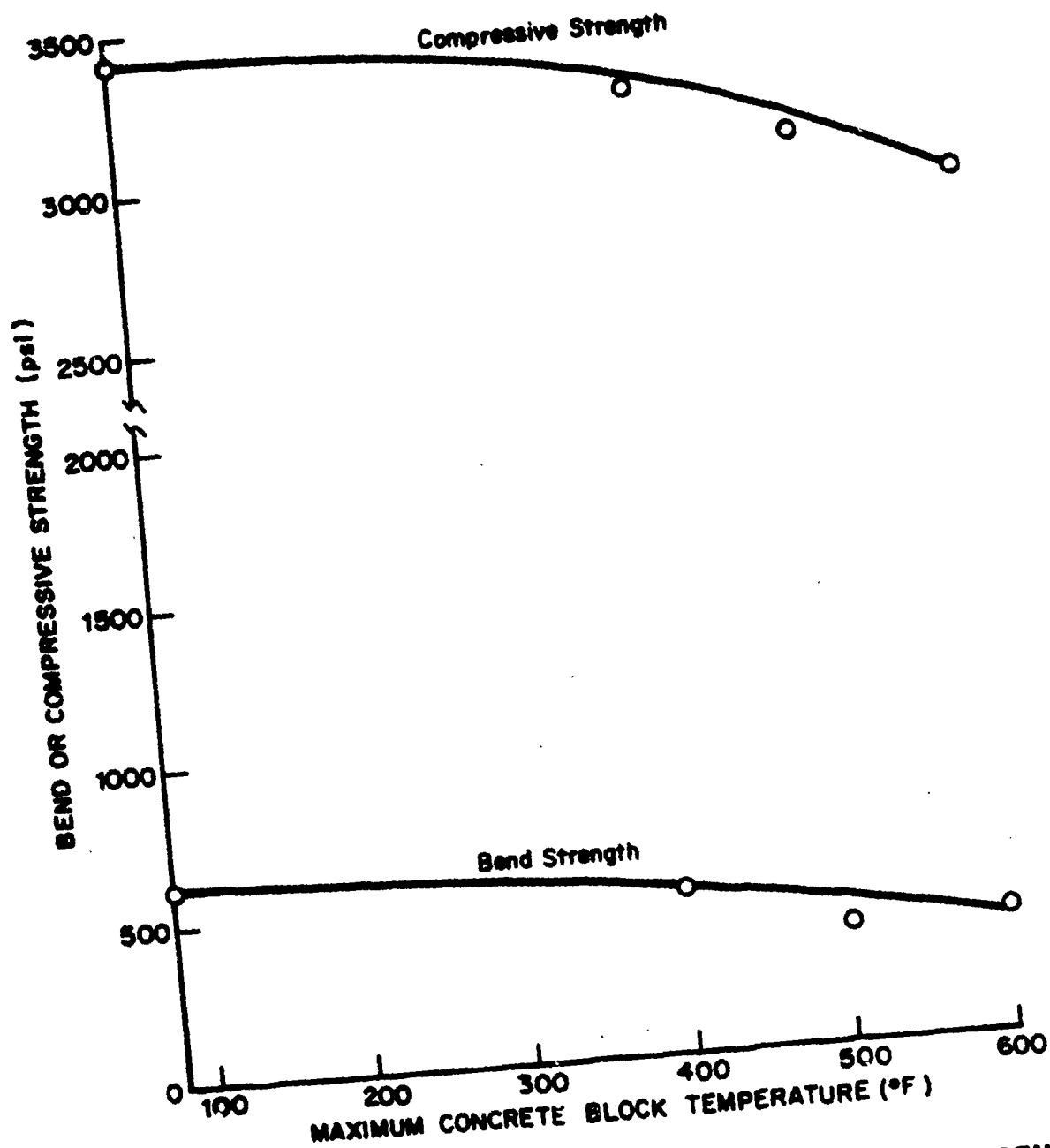
FIGURE 8-7 COMPARISON OF COMPRESSIVE AND BEND STRENGTH VALUES FOR OVEN HEATED NEW CONCRETE BLOCK

Source: Arthur D. Little, Inc.

Sample	Average Compressive Strength (psi)	Percent of Compressive Strength of Unheated Sample	Sample	Average Compressive Strength (psi)	Percent of Compressive Strength of Unheated Sample
Floor Unheated	3125	100.0	Lintel Unheated	5166	100.0
Floor Heated to 445°F	2388	76.4	Lintel Heated to 500°F	3726	72.1
			Lintel Heated to 580°F	3082	59.7

Examination of these floor and lintel test specimens revealed, however, that the aggregate used in these samples was much larger (up to 1/2 inch) than that in the new concrete block (up to 3/8 inch). The larger aggregate in these smaller test specimens may have influenced their compressive strength by creating relatively large weak spots in the cross section of the test specimen. (The ASTM procedure for determining compressive strength of cast concrete, ASTM C-42, requires a specimen diameter of at least 4 inches and at least three times the maximum size of the aggregate rather than the 2.4:1 ratio used in our test samples.)

8.3.4 X-ray Diffraction Analysis

Samples of the structural wall (west wall of Building 4L-5) were taken before and after heating and analyzed by X-ray diffraction analysis. A detailed analysis of spectra was not made but a comparison of the various peaks was made. There were no major differences between the heated and unheated samples indicating the loss or generation of any new compounds, but there was evidence of some minor peak shifting indicating the loss of chemically bound water. This water loss, which commences at 212°F, seems to parallel the loss in bend and compressive strength.

At temperatures over 212°F, the expansion of concrete paste and aggregate begins to be opposed by contraction of the concrete paste as it loses water. At approximatly 570°F, contraction of the concrete paste due to water loss actually exceeds the thermal expansion.[12,13] Because the

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[12]Harada, T., et al, 1972. "Strength, Elasticity and Thermal Properties of Concrete subjected to Elevated Temperatures." Concrete for Nuclear Reactors, Vol. 1, pp 377-406 SP34. American Concrete Institute, Detroit, Mich.

(Footnote Continued)

aggregate continues to expand over this entire temperature range, severe cracking is reported to occur.[14]

8.3.5 Significance of Structural Strength Loss in Thermally Decontaminated Buildings

The internal heating under controlled conditions of buildings requiring thermal decontamination will result in some loss of structural strength in non-combustible structural materials, such as concrete or concrete block. Building design is based, as a general rule, on allowable working stress levels in the structural members equal to one-fourth of the ultimate structural strength, or on 62% of the yield strength, whichever is lower. For concrete structures, the former limit normally would govern. It should be understood that this rather large factor of safety of four on primary stresses is somewhat misleading: in exchange for this large factor, building codes allow design stress analysis to be simplified. Thus, secondary stresses and local stresses are ignored, on the basis that, whatever their cause, the strength inherent in the structure due to the conservative design will be adequate. Accordingly, local stresses caused by, for example, holes cut into structural members to accommodate pipes, wires, ducts, etc. are not specifically analyzed. Nor are secondary stresses such as those developed by redundant structural members. Overall then, in local areas of the structure, the factor of safety may be considerably less than four but in terms of structural capacity to resist primary design loads the factor of safety would be close to four.

In some cases, the factor of safety for primary loads may be considerably more than four. Frequently, structural members (or, say, the thickness of concrete walls or concrete blocks) may be larger than necessary because of the availability of oversized structural components. If buildings of several different sizes are to be constructed at a given site, it may be more economical to acquire one size of concrete block whose width is adequate for the larger building. The smaller buildings would then be overdesigned when constructed with the same block size. (We do not know, for example, how much of a safety factor was used in the design of Building 4L-5 at Cornhusker AAP.)

Tests on new concrete blocks indicate that exposure to the thermal decontamination process would reduce the compressive strength of the building walls constructed of concrete block by 5 to 15% (Section 8.3.3). Concrete foundations and footings would, most likely, not be measurably

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(Footnote Continued)

[13] Zoldners, N.G., 1971. "Thermal Properties of Concrete Under Sustained Elevated Temperatures." *Temperature and Concrete* Publication SP-25, 1-32. American Concrete Institute, Detroit, Mich.

[14] Petterson, P.H., 1966. "Hardened Concrete -- Resistance to High Temperature." *Concrete and Concrete Making Materials* STP169-A. American Society for Testing Materials, Detroit, Mich.

affected because they would not reach the same temperatures as the walls. Thus, structures supported by concrete block materials would be unlikely to fail, as a result of the thermal decontamination; however, structures considered for reuse after decontamination would have to be evaluated to determine if the structure still meets local construction codes and safe design criteria.

The structural effects of thermal decontamination on existing buildings can be estimated based on the structural design and configuration of the building. For this, as-constructed structural drawings would be needed. Structural design calculations would also be helpful. If these are not available, then a detailed inspection of the specific buildings would be required.

Since many buildings of different sizes at several sites are involved, inspection of each building may not be practical. As an alternative, it may be possible to inspect, or review structural design information, of a representative selection of these buildings (or of the more important of the buildings) to develop some general guidelines for the expected effects of thermal decontamination. Furthermore, such guidelines could (and should) include criteria for acceptable uses of such buildings which would take into account the reduced capacity of the building's structure.

They could also provide guidance for the possible need and location of additional structural members to increase the structural capacity of portions of the building, if it appears that such strengthening would be desirable. An example would be the placement of interior lally columns (prior to the decontamination process) to provide added support to roof beams or rafter systems which may be affected by the decontamination process. Such additional structural members may also be needed as a retrofit following the thermal decontamination to increase the structural integrity of the building system.

8.4 Decontamination of Surfaces of Heated Concrete Blocks

Surface TNT contamination of the concrete blocks (taken from the sack sump cesspool of Building 4L-5) was measured before and after heating in the pilot tests using the acetonitrile/filter paper wipe procedure described in an earlier Arthur D. Little, Inc. Report[15]. These analyses were used to estimate residual levels and destruction efficiency of the surface TNT contamination. (The procedure used for analyzing these wipe samples is described in detail in Appendix A. The QA/QC procedures followed during the collection and chemical analysis of all the samples were described in our Pilot Plant Test Plan[16].)

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[15] Ibid Footnote 3

[16] Ibid Reference 4

Arthur D. Little, Inc.

Table 8-3 lists all of the wipe sample analyses. The effect of maximum heating temperature on destruction efficiency and residual surface contamination is summarized below:

Maximum Surface Temperature (°F)	Maximum Residual TNT Concentration (ug per sq cm)	TNT Destruction Efficiency (%)
>600	<0.18	99.9+
500-600	0.7	99.9+
400-500	1.02	98.7 - 99.9+
300-400	1.8 - 3.1	99.0 - 99.6

A careful examination of the data listed in Table 8-3 indicates that the lower destruction efficiencies calculated for block sufaces heated to 300 to 500°F (destruction efficiencies of less than 99.9%) may principally be due to the fact that most of these samples were much lower in initial TNT concentration. Thus, the real criteria for an acceptable treatment level may be residual concentration rather than destruction efficiency.

It is interesting to note, however, that the concentration of TNT was reduced to below the detectable level in all cases, even at a temperature as low as 400°F, when the blocks were sprayed with caustic.

8.5 Decontamination Within the Interior of Heated Concrete Blocks

Core samples taken from the concrete blocks before heating, and samples broken from the blocks after heating, were crushed and extracted. The extracts were analyzed by the procedures described in Appendices A and B, and the results of these analyses are listed in Table 8-4. For some of the samples heated to higher temperatures (over 600°F) the destruction efficiency appears to be lower (83 to 84%) than for samples heated to only 380 to 400°F (99+ %). Although these results may at first appear to be anomalous, it should be noted that the residual level of TNT in every case in the second pilot test was below the detectable level (<16.9 ug total or <0.11 ug/gm for a 150 gm block sample). With one exception, all the residual levels of TNT after heating in the first pilot test were also at or below the 0.11 ug/gm level. Again, as in the case of the surface decontamination efficiency, the value of percent internal destruction efficiency is determined primarily by the starting contaminant concentration, so the real measure of effective decontamination should, perhaps, be residual contamination level rather than destruction efficiency. In general, it appears that heat treatment of the TNT contaminated concrete blocks at a temperature of 400 to 645°F will reduce the internal residual TNT concentration to 0.16 ug/gm or less. In addition, based on surface sampling of the block (826 sq cm face) and the

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TABLE 8-3

SUMMARY OF CONCRETE BLOCK SURFACE DECONTAMINATION RESULTS

Pilot Test 1 (June 1986)

Sample Number Before	After	Filter Wipe	Concrete Block Side	Surface Sample* TNT Concentration Before	After	Destruct. Effic. (%)	Maximum Block Surface Temp. (°F)
4457†	4491	9-7F(FF)	FRONT	94000	<1.17**	>99.9	638
4458	-	9-7B(FB)	BACK	6230	-	-	-
4459†	4492	9-8F(GF)	FRONT	41500	<1.17	>99.9	642
4460	4493	9-8B(GB)	BACK	990	<1.17	>99.8	457
4461†	4485	9-9F(CF)	FRONT	12400	<1.17	>99.9	641
4462	4486	9-9B(CB)	BACK	1240	<1.17	>99.9	436
4463	4489	9-12F(EF)	FRONT	8230	<1.17	>99.9	630
4464	4490	9-12B(EB)	BACK	620	1.32	>99.8	414
4465†	4483	9-13F(BF)	FRONT	750	<1.17	>99.8	640
4466	4484	9-13B(BB)	BACK	340	1.29	99.6	437
4467†	4496	9-14F(IF)	FRONT	6830	<1.17	>99.9	642
4468	4497	9-14B(IB)	BACK	6480	3.23	>99.9	432
4469	4481	9-17F(AF)	FRONT	2260	2.12	>99.9	618
4470	4482	9-17B(AB)	BACK	1150	11.6	99.0	373
4471	4494	9-19F(HF)	FRONT	17100	<1.17	>99.9	645
4472	4495	9-19B(HB)	BACK	790	<1.17	>99.8	428
4473	4500	9-27F(KF)	FRONT	15300	<1.17	>99.9	615
4474	4501	9-27B(KB)	BACK	3600	14.1	99.6	332
4475†	4498	9-28F(JF)	FRONT	48400	<1.17	>99.9	640
4476	4499	9-28B(JB)	BACK	490	6.5	98.7	416
4477	4487	9-29F(DF)	FRONT	7700	<1.17	>99.9	640
4478	4488	9-29B(DB)	BACK	470	2.1	99.6	400
4479	4502	9-30F(LF)	FRONT	151000	<1.17	>99.9	635
4480	4503	9-30B(LB)	BACK	3770	19.7	99.5	371

Pilot Test 2 (October 1986)

Sample Number Before	After	Filter Wipe	Concrete Block Side	Surface Sample* TNT Concentration Before	After	Destruct. Effic. (%)	Maximum Block Surface Temp. (°F)
4642	4651	8-7	FRONT	123000	3.10	>99.9	571
4643	4652	7-30	FRONT	4540	<2.17**	>99.9	582
4644	4653	7-8	FRONT	3580	3.75	>99.9	560
4645	4654	7-2	FRONT	38400	4.39	>99.9	587
4646	4655	7-2	BACK	2730	2.55	>99.9	480
4647	4656	7-20	BACK	200	<2.17	>98.9	513
4648	4657	8-10B	BACK	450	<2.17	>99.5	382
4649	4658	7-9	BACK	330	2.42	99.3	409
4650	4659	8-27	BACK	240	<2.17	>99.1	437

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* ug TNT per 9 cm filter paper wipe. (To convert to ug TNT per sq cm concrete block surface at 10% recovery, divide by 6.36.)
** Limit of detection.   † Block surface caustic sprayed.

Source: Arthur D. Little, Inc.

Arthur D. Little, Inc.

TABLE 8-4

SUMMARY OF CONCRETE BLOCK INTERNAL DECONTAMINATION RESULTS[1]

Pilot Test 1 (June 1986)

Concrete Block	Block Side	Before Contamination Sample Number	Sample Weight (grams)	ug TNT	After Decontamination Sample Number	Sample Weight (grams)	ug TNT	Internal TNT Concentration (ug/gram) Before	After	Percent[2] Destruct. Effic.	Max. Block Surface Temp. (°F)
9-12(EF)	FRONT	4435	110.0	44.3	4447	132.6	9.2	0.40	.069	82.7	620
9-19(HF)	FRONT	4437	110.2	76.0	4449	152.7	16.7	0.69	0.110	83.2	645
9-14(IF)†	FRONT	4436	129.6	1760.0	4451	164.0	<9.2	13.60	<.056	>99.6	642
9-28(JF)†	FRONT	4439	105.3	718.0	4452	147.8	9.2	6.82	.062	99.1	640
9-27(KF)	FRONT	4438	116.8	1370.0	4453	94.8	24.6	11.80	.260	97.8	615
9-30(LF)	FRONT	4440	106.1	336.0	4455	118.7	<9.2	3.17	<.078	97.5	635

Pilot Test 2 (October 1986)

Concrete Block	Block Side	Before Contamination Sample Number	Sample Weight (grams)	ug TNT	After Decontamination Sample Number	Sample Weight (grams)	ug TNT	Internal TNT Concentration (ug/gram) Before	After	Percent[2] Destruct. Effic.	Max. Block Surface Temp. (°F)
8-7	FRONT	4610	102.1	109	4626	126.0	<16.9	1.06	<.134	>87.4	571
7-30	FRONT	4611	102.5	<16.9	4627	123.2	<16.9	<0.16	<.138	.	582
7-2	FRONT	4612	116.8	1390	4628	117.3	<16.9	11.90	<.144	>98.8	587
7-20	FRONT	4613	114.8	186	4629	227.5	<16.9	1.62	<.074	>95.4	581
7-29	FRONT	4614	86.7	479	4630	105.7	<16.9	5.52	<.160	>97.1	560
7-8	FRONT	4615	96.7	<16.9	4631	147.4	lost	<.18	not analyzed	.	560
8-10A†	FRONT	4616	122.6	683	4632	150.6	<16.9	5.57	<.112	>98.0	538
8-10B†	FRONT	4617	101.9	1130	4633	165.8	<16.9	11.10	<.102	>99.1	503
8-27	BACK	4618	94.1	<16.9	4634	134.7	<16.9	<0.18	<.126	.	437
7-9	BACK	4619	114.3	2680	4635	156.2	<16.9	23.40	<.108	>99.5	409
7-2	BACK	4620	122.8	138	4636	122.1	<16.9	1.12	<.139	>87.6	480
7-20	BACK	4621	111.9	77.3	4637	238.4	<16.9	0.69	<.071	>89.7	513
7-29	BACK	4622	108.8	<16.9	4638	140.5	<16.9	<0.16	<.121	.	506
7-8	BACK	4623	112.3	259	4639	125.9	<16.9	2.31	<.135	>94.2	480
8-10A	BACK	4624	114.1	436	4640	149.3	<16.9	3.82	<.114	>97.0	448
8-10B	BACK	4625	96.1	17500	4641	163.6	<16.9	182.00	<.104	>99.9	382

(1) Within the solid, concrete; portion of the concrete block.

(2) % Destruction Efficiency = $\left[\dfrac{(Z-Y)}{Z}\right]$ X 100   where: Z = concentration before decontamination (ug/g)

Y = concentration after decontamination (ug/g)

† Caustic sprayed blocks.

Source: Arthur D. Little, Inc.

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interior solids of the block (17,120 g per block), most of the initial TNT contamination of these blocks was at the block surface.

To gain some limited insight into the major types of TNT decomposition products that may result from this heat treatment process, it was decided to analyze the extract from one of the unpainted, contaminated concrete blocks. A sample of concentrated concrete block extract from block 7-2 (front) after heating (Sample 4628) was analyzed by GC/MS. The results are reported in Table 8-5. The major breakdown products of the TNT explosive in the block appear to be trinitrobenzene and various long chain alcohols, alkanes and amides.

8.6 Gas Sample Analyses

The filters and sorbents used in the VOST and MM5 sampling trains for sampling the gas stream from the test area (to the afterburner) and the hot gas stream leaving the afterburner were extracted and the extracts then analyzed as described in Appendix A. The extract analyses provided qualitative information on the types of compounds present in these gas streams and quantitative measurement of the concentration of hydrocarbons and TNT in these gas streams. All the samples were analyzed by GC, high performance liquid chromatography (HPLC) and selected samples were analyzed (qualitatively) by GC/MS.

8.6.1 HPLC Analyses of VOST Samples for Explosives - Pilot Test 1

Samples were obtained for analysis from three parts of the VOST sampling system: the particulate filters, the Tenax[8] adsorbent and the activated carbon adsorbent backup tubes. Only one filter sample of the VOST collected any measurable particulate. Sample train #3 filter was found to have collected approximately 0.83 ug of RDX per liter of gas sampled during the time period of 4 to 7 hours into the test run. No further evidence of the presence of RDX was found, however, in any other of the gas samples in either pilot test, so the origin of the RDX is not known.

In the first set of analyses of the extracts from the Tenax[8] adsorbent from the VOST sampling system, no measurable quantities of explosive were found. To increase the sensitivity of the analyses, the Tenax[8] adsorbent extracts (20 ml each) were concentrated and reanalyzed. The results are presented in Table 8-6. As can be seen from Table 8-6, there was a measurable quantity of TNT in the hot gas stream from the test area in the 7 to 10 hour time frame in the first pilot test that corresponded to an outlet gas stream temperature of 350 to 400°F. (This information was used in the second pilot test to determine the sampling time period with maximum TNT emissions for the MM5 train sampling of the gas stream entering and leaving the afterburner.)

Initial analysis of the activated carbon extracts did not show any indication of the presence of explosives. Further concentration and reanalysis of the activated carbon extracts also did not show any presence of explosives.

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TABLE 8-5

COMPOUNDS IDENTIFIED IN TNT CONTAMINATED CONCRETE BLOCK 7-2 AFTER HEATING
(Sample 4628)

M.W.	Compound	Mass on Column (ng)	Estimated Concentration (ug/g concrete)	Reportable Concentration
146	Dichlorobenzene	<1[b]	<0.01	Trace[a]
>120	Long Chain Alcohol	<1	<0.01	Trace[a]
>130	Long Chain Alcohol	<1	<0.01	Trace[a]
168	Dinitrobenzene	<<1[c]	<<0.01	Trace[a]
>200	Unknown	1	0.01	Trace[a]
213	Trinitrobenzene	1	0.01	Trace[a]
178	Phenanthrene	<1	<0.01	Trace[a]
183	Dinitrobenzamine?	<1	<0.01	Trace[a]
>220	Long Chain Alcohol	<1	<0.01	Trace[a]
242	Long Chain Alcohol	2	0.02	Trace[a]
252	Long Chain Alkene	1	0.01	Trace[a]
202	Pyrene or Fluoranthene	<1	<0.01	Trace[a]
204	Phenyl Naphthalene	<1	<0.01	Trace[a]
>200	Long Chain Amide	<1	<0.01	Trace[a]
>220	Long Chain Alkane	1	0.01	Trace[a]
281	Long Chain Amide	1	0.01	Trace[a]
>250	Long Chain Amide	1	0.01	Trace[a]
283	Long Chain Amide	1	0.01	Trace[a]
398	Ethanol,2 Butoxy, Phosphate?	1	0.01	Trace[a]
228	Chrysene or Triphenylene or Naphthacene	<1	<0.01*	Trace[a]
>300	Silylated Material	<1	<0.01	Trace[a]
>280	Long Chain Alkane	1	0.01	Trace[a]
>280	Long Chain Alkane	<1	<0.01	Trace[a]
>300	Silylated Material	<1	<0.01	Trace[a]
252	Benzopyrene or Benzo-fluoranthene	1	0.01*	Trace[a]

[a] Trace — Less than 0.1 ug/g concrete which would correspond to class 2 certification limit of 10 ug/mL in solvent extract

[b] <Less than

[c] <<Much less than

*Polyaromatic hydrocarbons suspect of carcinogenic potential for man by American Conference of Governmental Industrial Hygienists (ACGIH)

Source: Arthur D. Little, Inc.

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Table 8-6

HPLC ANALYSES OF CONCENTRATED VOST TENAX⬤ EXTRACT SAMPLES

Sample Lab No.	Tenax⬤ Field No.	Pilot 1 Test Run Time (hrs)	Extract Concentration Factor	Total TNT Mass (ug)	Volume Sampled (liters)	Building 4L-5 Gas Stream TNT Concentration (ug/L)
4409	1	1-4[1]	20	(0.14)[2]	42.8	0.0033
4410	3	4-7	30	<0.10	45.2	<0.0022
4411	3	7-10	20	0.42	51.7	0.0093
4412	4	10-13	20	<0.15	49.4	<0.0030
4413	5	13-16	20	<0.15	49.8	<0.0030
4414	6	16-19	30	<0.10	49.0	<0.0020
4415	7	19-21	20	<0.15	28.6	<0.0052
4416	8	21-23	30	<0.10	27.2	<0.0037
4417	9	23-25.5	30	<0.10	27.7	<0.0036
4418	10	25.5-27	20	<0.15	18.5	<0.0081
4419	11	27-28	10	<0.31	15.1	<0.020
4420	12	--	20	<0.15	0.00	--

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[1] Hours after starting up air heater (air heater started at 1300 hrs on 6/18/87).

[2] Value below Hubaux and Vost Detection limit of 0.15 mg but greater than lowest standard.

Source: Arthur D. Little, Inc.

🔺 Arthur D. Little, Inc.

8.6.2 GC Analyses of VOST Samples for (C_7 to C_{17}) Hydrocarbons - Pilot Test 1

Table 8-7 lists the total chromatographable organics (TCOs) extracted from each of the Tenax® adsorbent/activated carbon sample trains. (The TCOs are generally hydrocarbons with a chain length of 7 to 17 carbons.)

Figure 8-8 shows a plot of the sum of the TCOs from the Tenax® adsorbent and activated carbon in the test area exit gas stream versus time and temperature. Hydrocarbon (TCO) emissions from the test area climbed slowly until the block surface temperature reached about 500°F at which time they increased rapidly. When the air heater temperature was turned down near the end of the test, the emission level from the test area also dropped sharply showing the sensitivity of TCO emission level to temperature. (This temperature of over 500°F is most likely related to paint decomposition or paint component vaporization.)

8.6.3 Particulate Collected in MM5 (Afterburner Outlet) Sample - Pilot Test 1

A particulate weight of 1.5 mg was collected in the MM5 filter from the 4.55 cu m of afterburner outlet gas. (This is equivalent to 0.00014 gr/scfd which is well below the EPA particulate limitation - see Section 9.0.)

8.6.4 HPLC Analyses of MM5 (Afterburner Outlet) Samples for Explosives - Pilot Test 1

One MM5 sampling train was used in the first pilot run to measure stack (outlet) explosive emissions from the afterburner. Plant (fire hydrant) water was used as cooling water for the MM5 impinger train. Because the final impinger in this MM5 train fractured during the sampling and cooling water may have entered the sampling train, a water sample from the fire hydrant was also taken for analysis. Analyses of the extracts taken from the filter, XAD-2® adsorbent, and moisture condensate are presented in Table 8-8. The results presented in this table indicate a low level of TNT emissions from the afterburner during a time period when the VOST sampling was not indicating any measurable input of TNT to the afterburner (VOST samples 4 and 5). Again, as in the case of the RDX found in the second VOST train filter, the reason for presence of TNT in this sample is not clear.

8.6.5 GC Analysis of MM5 (Afterburner Outlet) Samples for TCOs - Pilot Test 1

The GC analysis of extract of the MM5 XAD-2® adsorbent showed a TCO concentration of about 0.7 ug per liter of gas in the afterburner outlet compared with the 40 ug per liter of gas in the afterburner inlet as measured by the VOST. This represents a reduction in TCO concentration of over 98%. Since a portion of this 0.7 ug per liter in the afterburner outlet may be attributable to unburned afterburner fuel, the reduction in the TCOs entering the afterburner may actually be greater.

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Table 8-7

GC ANALYSES FOR TCOs IN TEST AREA OUTLET GAS VOST SAMPLES

Sample Lab No.	Contaminated Block Surface Temperature (°F)	Field Number	Volume Sampled (liters)	Total Hydrocarbon Mass (ug)	Hydrocarbon Concentration (ug/L)
4409	--	1 Tenax®	42.8	422	9.86
4410	230	2 Tenax®	45.2	1020	22.6
4411	330	3 Tenax®	51.7	1040	20.1
4412	400	4 Tenax®	49.4	919	18.6
4413	440	5 Tenax®	49.8	2200	44.2
4414	470	6 Tenax®	49.0	780	15.9
4415	500	7 Tenax®	28.6	942	32.9
4416	520	8 Tenax®	27.2	885	32.5
4417	575	9 Tenax®	27.7	2420	87.4
4418	590	10 Tenax®	18.5	1890	102.0
4419	600	11 Tenax®	15.1	<414	<27.4
4420	--	12 Tenax®	0.00	<414	--
4421	--	1 Charcoal	42.8	<207	<4.84
4422	230	2 Charcoal	45.2	211	4.67
4423	330	3 Charcoal	51.7	865	16.7
4424	400	4 Charcoal	49.4	839	17.0
4425	440	5 Charcoal	49.8	<207	<4.16
4426	470	6 Charcoal	49.0	991	20.2
4427	500	7 Charcoal	28.6	913	31.9
4428	520	8 Charcoal	27.2	1210	44.5
4429	575	9 Charcoal	27.7	349	12.6
4430	590	10 Charcoal	18.5	258	13.9
4431	600	11 Charcoal	15.1	320	21.2
4432	--	12 Charcoal	0.00	<207	--

Source: Arthur D. Little, Inc.
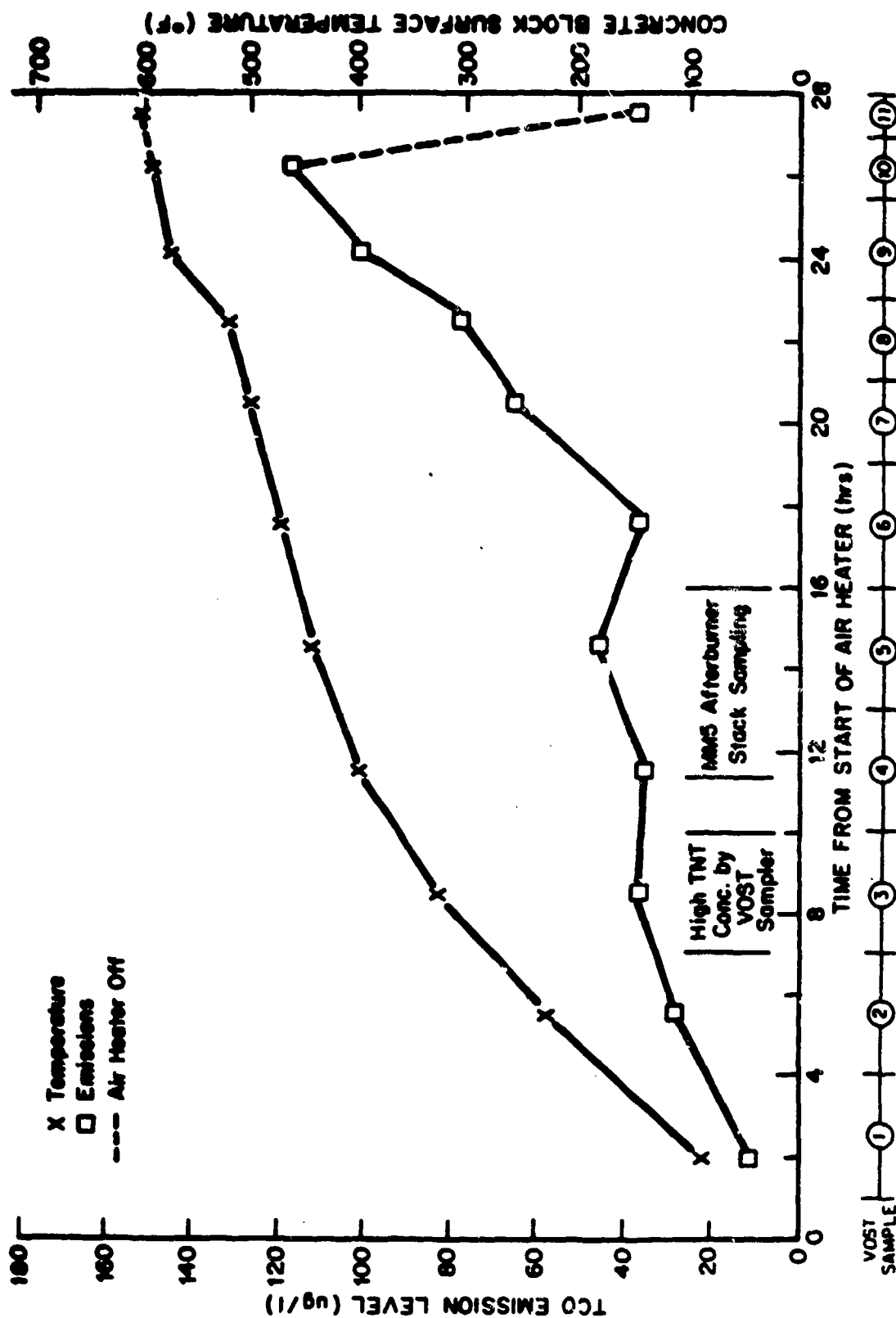
FIGURE 8-8 BUILDING 4L-8 TCO EMISSIONS vs TIME AND TEMPERATURE—PILOT TEST 1

Source: Arthur D. Little, Inc.

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▲ Arthur D. Little, Inc.

TABLE 8-8

HPLC ANALYSES OF MODIFIED METHOD 5 (____) SAMPLES

Pilot Test 1

Lab Number	Sample Description*	Mass (ug)		Volume Sampled (liters)	Concentration (ug/L)	
		TNT	RDX		TNT	RDX
4390	XAD-2® Trap	4.99	<0.66	4550	0.0011	<1.4×10^{-4}
4391	XAD-2® Blank	<0.41	<0.66	0.0	...	...
4392	Filter #62	<0.41	<0.66	4550	<9.0×10^{-5}	<1.4×10^{-4}
4393	Filter #63 Blank	<0.41	<0.66	0.0	...	...
4394	Condensate	<0.41	<0.66	4550	<9.0×10^{-5}	<1.4×10^{-4}
4395	Hydrant Water	<0.41	<0.66	...	...	...
4396	Milli-Q Water Blank	<0.41	<0.66	0.0	...	...

*These samples were taken during the time period 11.25 to 15.75 hrs after startup of the air heater in Pilot Test 1.

Source: Arthur D. Little, Inc.

8.6.6 Gravimetric Analyses of MM5 (Afterburner Outlet) Sample for High Boiling Organics - Pilot Test 1

A portion of the organics collected by the XAD-2' sorbent were high boiling organics (bp over 300°C) not measurable by GC. The weight of these organics is estimated by evaporation of an aliquot of the XAD-2' extract. A weight of approximately 10.1 mg of these high boiling organics was collected from the 4.55 cu m afterburner outlet gas sample. This high boiling organic fraction also contains some Principal Organic Hazardous Constituents (POHCs) which are regulated by the EPA (see Section 8.6.7).

8.6.7 GC/MS Analysis of MM5 (Afterburner Outlet) Sample - Pilot Test 1

The extract of the XAD-2' adsorbent from the afterburner outlet MM5 sample was subjected to GC/MS analysis according to the procedures described in Appendix A. The compounds detected in the afterburner outlet in this pilot test are summarized in Table 8-9. One of the compounds listed (marked with an asterisk) is a suspect carcinogen but all the compounds in this table are only present in the parts per billion (ppb) range in the afterburner outlet gas stream. A few of these compounds listed in Table 8-9 are also on EPA's list of POHCs[17]. (This point will be discussed in greater detail in Sections 8.6.8 to 8.6.11.) It should also be noted that the number of compounds and the concentrations of these potentially carcinogenic materials in this afterburner outlet sample are less than those cited later in Section 8.6.9 of this report for the afterburner outlet samples in Tables 8-13 and 8-14.

8.6.8 HPLC Analyses of MM5 (Afterburner Inlet and Outlet) Samples for Explosives - Pilot Test 2

In the second pilot run, two MM5 trains were used to sample the gas stream from the test area (to the afterburner) and one MM5 train was used to sample the afterburner outlet gas stream. The gas stream leaving the test area was sampled during the two time periods when: (1) maximum TNT emissions were expected; and (2) maximum TOC emissions were anticipated. The gas stream leaving the afterburner was sampled simultaneously with the maximum inlet TNT emissions (Refer to Table 7-2) to allow TNT destruction efficiency (DRE) to be estimated for the afterburner. (The afterburner was operated at 2000°F in the first pilot test and 1800°F in the second pilot test.)

The results of the HPLC analyses of extracts from the MM5 train components for the second pilot test are summarized in Table 8-10. As predicted, the time at which the MM5 sample was taken from the afterburner stack coincided with the afterburner inlet sample with the highest TNT concentration. The TNT destruction efficiency achieved in the afterburner, as measured by these two MM5 trains, was about 99.7%.

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[17] Federal Register 40 CFR Part 261 Appendix VIII

/⚮ Arthur D. Little, Inc.

TABLE 8-9

TENTATIVELY IDENTIFIED COMPOUNDS IN AFTERBURNER OUTLET MM5 SORBENT (4390) - PILOT TEST 1

M.W.	Compound	Estimated Concentration (ug/cu m)	Reportable Concentration
100	Methylpentanol	15.2	Trace[a]
92	Toluene	3.2	Trace[a]
164	Phthalate	3.2	Trace[a]
>200	Silicone	2.8	Trace[a]
100	Methylcyclopentanol	2.6	Trace[a]
136	Dichlorocyclopentane	2.4	Trace[a]
106	Benzaldehyde	5.1	Trace[a]
>200	Silicone	1.7	Trace[a]
>280	Silicone	6.4	Trace[a]
120	1-phenylethanone	4.1	Trace[a]
152	Dichlorocyclohexane	3.9	Trace[a]
126	Methyl ester of hexadienoic acid	4.3	Trace[a]
134	Ethylbenzaldehyde	8.8	Trace[a]
134	Ethylbenzaldehyde (isomer)	4.1	Trace[a]
128	Naphthalene	2.8	Trace[a]
>260	Silicone	2.4	Trace[a]
135	Benzothiazole	3.6	Trace[a]
>170	Hydrocarbon	1.5	Trace[a]
148	Ethylphenylethanone	3.2	Trace[a]
148	Dimethylphenylethanone	1.7	Trace[a]
184	Hydrocarbon (C_{14})	3.9	Trace[a]
>300	Silicone	2.1	Trace[a]
162	Phenylene ethanone	2.4	Trace[a]
198	Hydrocarbon (C_{15})	9.2	Trace[a]
>300	Silicone	3.4	Trace[a]
>190	Fluorinated/Chlorinated Hydrocarbon	2.1	Trace[a]
220	Dimethyl Cyclohexadienedione	3.6	Trace[a]
>200	Hydrocarbon	3.0	Trace[a]
>200	Hydrocarbon	3.9	Trace[a]
>280	Silicone	2.8	Trace[a]

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TABLE 8-9

TENTATIVELY IDENTIFIED COMPOUNDS IN AFTERBURNER OUTLET MM5 SORBENT (A380) - PILOT TEST 1

M.W.	Compound	Estimated Concentration (ug/cu m)	Reportable Concentration
>200	Hydrocarbon	6.0	Trace[a]
>300	Silicone	3.0	Trace[a]

(structure diagram of benzene ring with C-NH (C4H9)2 and CH3 substituent)

M.W.	Compound	Estimated Concentration (ug/cu m)	Reportable Concentration
191	N-(1,1-dimethylethyl)-4-methylbenzamide	5.4	Trace[a]
222	Phthalate	13.7	Trace[a]
286	Dimethylethyl, ethylopropanoic acid	9.6	Trace[a]
196	Phenylmethyldimethylbenzene	13.5	Trace[a]
>220	Hydrocarbon	8.6	Trace[a]
236	Fused ring aromatic - methyl substituted (MW236)	6.2	Trace[a]
>300	Silicone	17.4	Trace[a]
>230	Hydrocarbon	10.7	Trace[a]
178	Phenanthrene	10.3	Trace[a]
188	Internal Standard (D_{10}anthracene)	11.4	Trace[a]
>230	Hydrocarbon	9.4	Trace[a]
>240	Hydrocarbon	5.4	Trace[a]
>240	Hydrocarbon	7.9	Trace[a]
>240	Hydrocarbon	8.8	Trace[a]
304	Phthalate	10.3	Trace[a]
>300	Silicone	10.3	Trace[a]
>300	Phthalate	8.1	Trace[a]
228	*Chrysene	4.5	Trace[a]
>300	Silicone	4.5	Trace[a]
>300	Silicone/co-eluting with an unknown chlorinated hydrocarbon	3.0	Trace[a]

[a]Trace - Less than 20 ug/cum which would correspond to Class 2 certification limit of 10 ug/mL in solvent extract.

*Polyaromatic hydrocarbon suspect of carcinogenic potential for man by ACGIH.

Source: Arthur D. Little, Inc.

TABLE 8-10

HPLC ANALYSES OF TNT IN MM5 SAMPLES OF
GAS ENTERING AND LEAVING THE AFTERBURNER - PILOT TEST 2

MM5 Train (All Components)	Weight of TNT (ug)	Gas Volume (cu m)	Gas Conc. (ug/cu m)	Destruction[a] Efficiency (%)
Stack 1	38.4	5.5026	6.98	99.7
Inlet 1	15830	6.095	2600	----
Inlet 2	3233	6.226	519	----

****

MM5 Train	Matrix	Sample No.	TNT (ug)	Volume (cu m)	Conc. (ug/cu m)
Stack 1	XAD-2	4587	<1.13	5.5026	<.205
Inlet 1	XAD-2	4588	12300	6.095	2010
Inlet 2	XAD-2	4589	3120	6.226	502
Field Blank	XAD-2	4591	<1.13	5.9412[b]	<.190
Stack 1	Filter	4592	<1.13	5.5026	<.205
Inlet 1	Filter	4593	<1.13	6.095	<.185
Inlet 2	Filter	4594	<1.13	6.226	<.181
Field Blank	Filter	4595	<1.13	5.9412	<.190
Stack 1	Condensate	4596	38.4	5.5026	6.98
Inlet 1	Condensate	4597	3530	6.095	579
Inlet 2	Condensate	4598	113	6.226	18.1
Field Blank	Condensate	4599	<1.13	5.9412	<.190

[a] % Destruction Efficiency $= \left[\dfrac{Z - Y}{Z} \right] \times 100$
where Z = concentration in inlet 1
where Y = concentration in stack 1

[b] Average of three MM5 train volumes

Note: 1000 ug/cu m = 0.1 ppm (vol/vol) TNT

Source: Arthur D. Little, Inc.

Arthur D. Little, Inc.

struction efficiencies of 99.99% are typically required by EPA for POHCs in hazardous waste incineration[18]. (A 99.9999% DRE is required for certain very thermally stable hazardous wastes.) These DREs are usually based on a relatively concentrated waste stream, however. For example, incineration of 0.1% solution of TNT (1000 ppm) in solvent with 25% excess air and a 99.99% destruction efficiency would yield an incinerator exhaust stream containing 0.002 ppm (vol) or 0.02 ug/L (20 ug/cu m) of TNT. This would be about three times the TNT concentration we measured in the afterburner exhaust in our pilot test.

In a recent report to EPA (from Arthur D. Little, Inc.), it was recommended that destruction efficiency not be measured for POHCs present in wastes in concentrations of less than 1000 ppm because of analytical difficulties in verifying DREs at these low concentrations[19].

Thus, although the DRE of TNT measured in our (second) pilot test was very slightly below the EPA requirement for POHC destruction, the concentration of TNT in the afterburner stack was also below that which would occur if a waste containing the minimum concentration of TNT recommended for regulation were burned.

8.6.9   GC Analyses of MM5 (Afterburner Inlet and Outlet) Samples for TCOs - Pilot Test 2

Solvent extracts of the components of the three MM5 trains were analyzed using gas chromatography to determine the concentration of TCOs (C_7 to C_{17} with boiling points of 100 to 300°C). The concentration of these light hydrocarbons (TCOs) in the two inlet gas streams and one afterburner gas outlet stream are summarized in Table 8-11. Destruction efficiency of these light (TCO) hydrocarbons was measured at about 92% as reported in Table 8-11. Although this destruction efficiency for these low boiling hydrocarbons may be used as an indicator of general incinerator (afterburner) performance, it does not have great significance from a regulatory standpoint. These low carbon number compounds are not usually found to have any serious health effects and the low total level of hydrocarbon emissions from this system would be unlikely to have any significant impact on ambient air quality or requirement for either Federal or state regulation.

8.6.10   Gravimetric Analysis of MM5 (Afterburner Inlet and Outlet) Samples for High Boiling Organics - Pilot Test 2

Some of the organics extracted from the three MM5 train components had boiling points of over 300°C and could not readily be quantified by gas

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[16]Federal Register 40 CRF Part 264.34

[19]Cerandulo, et al., "Sampling and Analysis Methods for Hazardous Waste Combustion" (2nd Edition) Report to EPA under Contract No. 68-02-3111 (Tech. Dir. No. 124) by Arthur D. Little, Inc. (February 1986).

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TABLE 8-11

GC ANALYSES OF HYDROCARBONS (TCOs) IN MM5 SAMPLES OF GAS ENTERING AND LEAVING THE AFTERBURNER - PILOT TEST 2

MM5 Train (All Components)	Weight of Hydrocarbons (mg)	Gas Volume (cu m)	Gas Conc. (mg/cu m)	Destruction[a] Efficiency (%)
Stack 1 (4.4 to 9.4 hrs of test)[b]	2.34	5.5026	0.425	92.2
Inlet 1 (4.4 to 9.4 hrs of test)	33.2	6.095	5.45	----
Inlet 2 (17.75 to 27.25 hrs of test)	39.3	6.226	6.31	----

****

MM5 Train	Matrix	Sample No.	Hydrocarbons (mg)	Volume (cu m)	Conc. (mg/cu m)
Stack 1	XAD-2	4587	1.85	5.5026	0.336
Inlet 1	XAD-2	4588	22.5	6.095	3.69
Inlet 2	XAD-2	4589	38.0	6.226	6.10
Field Blank	XAD-2	4591	0.116	5.9412[c]	.0195
Stack 1	Filter	4592	<.0703	5.5026	<.0128
Inlet 1	Filter	4593	<.0703	6.095	<.0115
Inlet 2	Filter	4594	<.0703	6.226	<.0113
Field Blank	Filter	4595	<.0703	5.9412	<.0118
Stack 1	Condensate	4596	0.494	5.5026	0.0898
Inlet 1	Condensate	4597	10.7	6.095	1.76
Inlet 2	Condensate	4598	1.3	6.226	0.209
Field Blank	Condensate	4599	<.0703	5.9412	<.0118

---

[a] % Destruction Efficiency $= \left[\dfrac{Z - Y}{Z}\right] \times 100$
where Z = concentration in inlet 1
where Y = concentration in stack 1

[b] Hours after starting afterburner
[c] Average of three MM5 train volumes

Source: Arthur D. Little, Inc.

△ Arthur D. Little, Inc.

chromatographic analysis. Consequently, one ml samples of each 10 mL extract were evaporated to determine the weight of higher boiling organics (over 300°C) present in each of the extracts. The results of these analyses and their conversion to concentration in the gas streams is presented in Table 8-12. Some high boiling organics were measured in the field blanks also, indicating a potential (low level) contamination of the extracting solvent. Thus, the hydrocarbon level found in the blank sample was subtracted from the values determined for the XAD-2, filter and condensate samples in calculating gas stream concentration and destruction efficiency. The concentration of these higher boiling hydrocarbons is of much greater concern than the concentration of the lower boiling hydrocarbons. This is due to the fact that the higher molecular weight compounds, particularly the polyaromatic hydrocarbons or polycyclic organic materials (POMs), include some compounds such as chrysene which is listed by ACGIH as being suspect of carcinogenic potential for man. Although emissions of some of these specific compounds in this category are being regulated, emissions of POMs in general are not currently being regulated except in regard to some specific industries (e.g., coal tar products). A number of the high boiling organic present in the afterburner inlet and outlet gases have been tentatively identified by GC/MS (see Section 8.6.11).

A problem was also encountered with the filters in each of the MM5 trains. All three filter discs were found to be stuck to the O-ring seal of the filter holder and part of each filter was lost in removing it from the filter holder. Although the two inlet sampling filters were somewhat blackened in color, their apparent weight after the test (and extraction of explosives and organics) was less than the original weight, probably due to the slight tearing of the filters in their removal from the filter holder. Thus, while the chemical analysis of organics and explosives from the filters is believed to be reliable, we have no measure of the weight of black material on the filter pad. (We believe the black material to be either carbon or particles of charred paint.)

8.6.11 GC/MS Analyses of MM5 (Afterburner Inlet and Outlet) Samples - Pilot Test 2

Because it was not possible to obtain a sufficiently concentrated sample of adsorbent extract in the VOST sampling system in the first pilot run to allow identification by GC/MS of the components in the gas stream leaving the test area, two MM5 trains were used in the second pilot test in place of the VOST sampling system. A 3 ml aliquot of the XAD-2 adsorbent extract from each of the two inlet MMS sampling trains was used for GC//MS analysis by the procedures described in Appendices A and B. The list of compounds from these two inlet MM5 trains and their relative concentrations is given in Tables 8-13 and 8-14.

In retrospect, it would have been useful to obtain an MM5 sample from the afterburner stack at the same time that the second afterburner inlet sample was taken (TCO and high boiling hydrogen emissions from the building to the afterburner inlet were maximum at that time). This would have allowed afterburner organic emissions to be measured under maximum inlet test conditions. Unfortunately, equipment limitations allowed sampling across

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TABLE 8-12

GRAVIMETRIC ANALYSES OF HIGH BOILING HYDROCARBONS IN MM5 SAMPLES OF GAS ENTERING AND LEAVING THE AFTERBURNER - PILOT TEST 2

Sample No.	Sample Description	Extract Residue Weight (mg/sample)	Gas Stream Conc. (mg/cu m)
4587	Stack 1 XAD-2	3.0	---
4588	Inlet 1 XAD-2	87.0	13.8
4589	Inlet 2 XAD-2	139.0	21.8
4591	Field Blank XAD-2	2.8	---
4592	Stack 1 Filter	0.5	---
4593	Inlet 1 Filter	1.5	---
4594	Inlet 2 Filter	2.0	---
4595	Field Blank Filter	2.5	---
4596	Stack 1 Condensate	3.0	---
4597	Inlet 1 Condensate	23.3	3.7
4599	Field Blank Condensate	0.5	---

% Destruction Efficiency

$$= \frac{\text{Total Inlet} - \text{Total Outlet}}{\text{Total Inlet}} \times 100$$

$$= \frac{[(87.0-2.8) + (23.3-0.5)] - [(3.0-2.8) + (3.0-0.5)]}{(87.0-2.8) + (23.3-0.5)} \times 100$$

$$= 97.5\%$$

Source: Arthur D. Little, Inc.

A. Arthur D. Little, Inc.

TABLE 8-13

Molecular Wt.	Compound	Gas Concentration (ug/cu m)
106	Benzaldehyde	20
140 (E)	Hydrocarbon	trace[a]
94	Phenol	20
126 (E)	Hydrocarbon	trace
>100	Hydrocarbon	trace
>250	Silylated Material/Mixture	trace
>280	Silylated Material	60
150 (E)	Hydrocarbon	trace
120	Phenyl Ethanone	20
154	Hydrocarbon	trace
154 (E)	Hydrocarbon	trace
142	Nonanone	trace
130 (E)	Heptanoic Acid	20
152	Hydrocarbon-Branched	trace
>260	Silylated Material	trace
134	Ethyl Benzaldehyde	trace
134	Dimethyl Benzaldehyde	30
122	Ethyl Phenol	trace
128	Naphthalene	trace
>300/140 (E)	Silylated Material/ Hydrocarbon Mixture	trace
122	Benzoic Acid	40
148	Hydrocarbon	trace
156	Dihydropentylfuranone	trace
184 (E)	Methyldodecane	trace
148	Ethyl Phenyl Ethanone	trace
168	Tetrahydropyranone	trace
148	Ethyl Phenyl Ethanone	trace
170	Hydrocarbon	trace
134	Isobenzofuranone	trace
148 (E)	Hydrocarbon	20

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TABLE 8-13 (Continued)

TENTATIVELY IDENTIFIED COMPOUNDS IN AFTERBURNER INLET SAMPLE 1
XAD SORBENT (4588) - PILOT TEST 2

Molecular Wt.	Compound	Gas Concentration (ug/cu m)
154	Hydrocarbon	trace
156	Dihydropentyl Furanone	trace
150 (E)	Branched Acid	trace
>300	Silylated Material	30
170	Decanoic Acid	trace
156	Decanone	trace
>180	Hydrocarbon	trace
182	1,3-Dinitro Toluene (isomer)	trace
198	Hydrocarbon	20
>300	Silylated Material	trace
176	Phenylhexanone	trace
220	Dimethylcyclohexadienedione	trace
186	Long Chain Acid	trace
182	DNT (targeted compound)	30
212	Branched Hydrocarbon	trace
170	Trimethylnaphthalene	trace
182	3,5-Dinitrotoluene	trace
212	Branched Hydrocarbon	40
>300	Silylated Material	70
191	Dimethyl Ethyl Benzamide	40
222	Phthalate	30
200	Dodecanoic Acid	30
226	Branched Hydrocarbon	trace
226	Branched Hydrocarbon	trace
198 (E)	Long Chain Acid	trace
224	Hydrocarbon	trace
224	Cyclohexadecane	trace
226	Hydrocarbon	50
210	TNT (targeted compound)	30
252	Branched Hydrocarbon	trace
254	Branched Hydrocarbon	30

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TABLE 8-13 (Continued)

TENTATIVELY IDENTIFIED COMPOUNDS IN AFTERBURNER INLET SAMPLE 1
XAD-SORBENT (A588) - PILOT TEST 2

Molecular Wt.	Compound	Gas Concentration (ug/cu m)
232	Hydrocarbon	trace
>300	Silylated Material	50
268	Branched Hydrocarbon	20
178	Phenanthrene	20
228/188	Coelution: Long Chain Acid/ Internal Standard	30
232	Hydrocarbon	30
>250	Phthalate	trace
254	Hydrocarbon	20
270	Hydrocarbon	20
290 (E)	Phthalate	30
>300	Silylated Material	40
192	Methyl Phenanthrene	trace
268	Hydrocarbon	20
278 (E)	Phthalate	30
256	Long Chain Acid	30
242	Phenyl-bicyclohexyl	trace
>250	Long Chain Ester	trace
>300	Silylated Material	40
270	Tetrahydro Indacene Dione	trace
202	Pyrene	20
196	Hydrocarbon	trace

[a]Trace - <16 ug/cu m - USATHAMA Class 2 Certified Reporting Limit.

(E) - Estimated

Source: Arthur D. Little, Inc.

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TABLE 8-14

TENTATIVELY IDENTIFIED COMPOUNDS IN AFTERBURNER INLET SAMPLE 2
XAS SORBENT (4589) - PILOT TEST 2

Molecular Wt.	Compound	Gas Concentration (ug/cu m)
106	Benzaldehyde	40
>100	Branched Hydrocarbon	20
94	Phenol	50
140 (E)	Hydrocarbon	20
100	Pyranone - saturated	40
120	Branched Hydrocarbon/Cumene	30
122	Hydroxy Benzaldehyde	30
>280	Silyated Compound	60
120	Phenyl Ethanone	30
168 (E)	Hydrocarbon - unsaturated	20
136	Benzoic acid-methyl ester	trace[a]
142	Nonanone	20
139	Nitro-phenol	30
156	Hydrocarbon	20
>280	Silylated Material	20
134	Ethyl Benzaldehyde	20
128	Ethyl Pyranone	trace
134	Dimethyl Benzaldehyde	trace
122	Ethyl Phenol	20
128	Naphthalene	trace
144	Nonanol	trace
122	Benzoic Acid	20
>300	Silylated Material	30
148	Ethanone-Ethylphenyl	20
148	Isobenzofurandione?	30
148	Benzenedicarboxylic Acid	40
172 (E)	Decanoic Acid?	30

TABLE 8-14 (Continued)

TENTATIVELY IDENTIFIED COMPOUNDS IN AFTERBURNER INLET SAMPLE 2 XAD SORBENT (4589) - PILOT TEST 2

Molecular wt.	Compound	Gas Concentration (ug/cu m)
126 (E)	Alcohol-long chain	trace
134	Benzenedicarboxaldehyde	30
184	Hydrocarbon	trace
>150	Hydrocarbon	trace
148	Benzopyranone	trace
178	Benzoic Acid Ester	40
162	Benzofuranone	40
150	Benzoic Acid Ester	trace
136	Benzenemethanol-Dimethyl	30
161	Indole-dione???	30
178	Benzoic Acid Ester	20
162	Ethanone-Phenylene (bis)	20
180 (E)	Hydrocarbon	trace
198	Hydrocarbon	20
147	Indole-dione??	30
147	Indole-dione??	30
147	Indole-dione??	30
186	Undecanoic Acid	20
196	Branched Hydrocarbon	20
139	Nitrophenol	40
>300	Silylated Material	60
222	Phthalate	20
200 (E)	Long Chain Fatty Acid??	30
226 (E)	Hydrocarbon	30
227	TNT	40
>250	Hydrocarbon	trace
>220	Hydrocarbon	30
196 (E)	Hydrocarbon Unsaturated	trace

8-45

TABLE 8-14   (Continued)

TENTATIVELY IDENTIFIED COMPOUNDS IN AFTERBURNER INLET SAMPLE 2
XAD SORBENT (4589) - PILOT TEST 2

Molecular Wt.	Compound	Gas Concentration (ug/cu m)
226	Pentadecanone	20
180 (E)	Benzoic Acid Ester	trace
>280	Silylated Material	trace
>300	Silylated Material	50
268	C_{19} Hydrocarbon-Branched	20
180	$C_{14}H_{12}$ Aromatic	trace
188	D_{10} Anthracene I.S.	trace
228	Tetradecanoic Acid?	30
278 (E)	Phthalate	trace
254	C_{18} Hydrocarbon-straight	20
228/204 (E)	Hydrocarbon (+) Phenylnaphthalene	20
>278 (E)	Phthalate	20
>300	Silylated Material	40
254 (E)	Long Chain Ketone	trace
197	Methanone, Diphenyl Oxime?	trace
268 (E)	Hydrocarbon	trace
278 (E)	Phthalate	trace
204	Phenyl-naphthalene	trace
236 (E)	Long Chain Ester?	30
282 (E)	Hydrocarbon	trace
202	Pyrene	trace
>300	Silylated Material	70
270	Phenanthrene-partially saturated	trace
270 (E)	Hydrocarbon	trace
270 (E)	Hydrocarbon	20
284	Octadecanoic Acid?	trace
>300	Silylated Material	30
310 (E)	Hydrocarbon	trace

TABLE 8-14 (Continued)

TENTATIVELY IDENTIFIED COMPOUNDS IN AFTERBURNER INLET SAMPLE 2
MM5 SORBENT (4589) - PILOT TEST 2

Molecular Wt.	Compound	Gas Concentration (ug/cu m)
300	Hexadecanoic Acid-Hydroxyl Ethyl Ester	trace
>300	Hydrocarbon	trace
>300	Hydrocarbon	trace
>300	Hydrocarbon	trace
>300	Silylated Material	30
>300	Silylated Material	30
>300	Phthalate	trace
>300	Silylated Material	20

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[a]Trace - <16 ug/cu m - USATHAMA Class 2 Certified Reporting Limit

(E) - Estimated

Source: Arthur D. Little, Inc.

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the afterburner (simultaneous inlet and outlet MM5 sampling) for only one
time period, during maximum inlet TNT emissions. In future pilot testing,
however, it would be desirable to measure afterburner emissions during
maximum hydrocarbon inlet conditions as well.

9.0 IMPLICATIONS OF TEST RESULTS REGARDING ENVIRONMENTAL REGULATIONS

Our pilot plant testing efforts to date, have indicated that heating the explosive contaminated concrete block building walls to 400 to 500°F will reduce the surface TNT concentration to about 1 ug/sq cm. Heating the blocks to 500 to 600°F will reduce the surface concentration to about 0.7 ug/sq cm while heating to over 600°F reduces the surface concentration to about 0.3 ug/sq cm. Similarly, decontamination of the interior of the concrete blocks by heating to over 400°F reduces the internal concentration of TNT to below the detectable limit (equal to about 0.11 ug/gm of concrete block).

Unfortunately, acceptable residual levels of explosives (TNT, DNT or RDX) on building materials have not, to our knowledge, been set by either the U.S. Army or the EPA. Consequently, how clean is clean enough is still an unanswered question. If residual TNT concentrations of 1 ug/sq cm at the surface and 0.1 ug/gram of block within the block are acceptable from an environmental or health standpoint, then the structural weakening of the building could be minimized by limiting the interior wall surface temperature to 500°F and the exterior wall surface to about 400°F. This would result in only a 5% loss in compressive strength compared to a 15% loss if the wall were heated to 500 to 600°F. [At 400 to 500°F, the loss of tensile (bend) strength would be limited to about 20%.] This limited loss in compressive and tensile strength would be unlikely to render the building unusable, particularly if subsequent reinforcing were used.

With respect to the potential for hazardous constituents being present in the building off-gas, it was noted that the highest concentrations of principal organic hazardous constituents (POHCs) in the gas leaving the building and entering the afterburner were TNT and TNT decomposition products at concentrations of up to 14 ppb as determined by GC/MS. The destruction efficiency (DRE) of the TNT vapors in the afterburner (as measured by HPLC analysis of XAD sorbent extract) was slightly over 99.7%. Destruction efficiency of the higher boiling hydrocarbons as a group (which includes TNT, TNT by-products and most of the other POHCs) was estimated at over 97.0% as measured at one second retention time (half way through the afterburner) at 1800°F. This is lower than the 99.99% DRE normally required for POHCs by EPA, but the specifically identified POHCs were only present in the afterburner inlet at 1 to 10 ppb and in the afterburner outlet at 1 to 2 ppb which are too low in concentration to allow accurate estimation of destruction efficiency. In any case, EPA regulation of these compounds at such low concentrations may eventually be determined based on ambient air concentration modelling rather than destruction efficiency, similar to the procedure being proposed for municipal waste incinerators.[20] In addition, it is generally accepted that there is a 1×10^5 dilution of stack gases in mathematical modelling of ground level ambient air

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[20]Harris et al., "Municipal Waste Combustion Study - Sampling Study," Draft Report to EPA Air and Energy Research Laboratory, RTP Contract No. 68-01-7266, by Arthur D. Little, Inc. (January 1987).

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concentration[21] which, in turn, would produce extremely low ambient air concentrations.

It is also of interest to note that the greatest volume of hydrocarbon emissions occurred late in the pilot test, when higher temperatures (over 500°F) were reached and after TNT emissions had declined, which indicates that most of the hydrocarbon emissions were due to pyrolysis of the paint. These pyrolysis products can be expected to be quite different in chemical nature than the explosive (TNT) decomposition products. Limiting heat treatment to a wall surface temperature of about 500°F should, therefore, not only reduce structural damage, but also reduce hydrocarbon emissions.

With respect to estimating particulate emissions, it should be noted that some problems were encountered in the particulate sampling. The concentration of particulate to or from the afterburner in the second pilot test is not known because of the damage to the filter pads during recovery. There was some darkening of the filter pads but no weight increase was measured. However, there was no visible stack opacity during the second pilot test and most of the burned paint from the test area walls that could have contributed to particulate emissions appeared to have either remained on the walls or fallen to the floor of the test area. In the first pilot test, the particulate collected in the MM5 filter amounted to only 1.5 mg for 4.55 cu m of gas sample from the afterburner outlet. This would be equivalent to 0.00014 gr/scfd and is well below the EPA particulate limitation for hazardous waste incineration of 0.08 gr/scfd.[22] It appears, therefore, that there is not a significant particulate release problem. Additionally, a decontamination temperature of about 500°F and the lower gas spacial velocities in a larger building would further reduce the potential for paint pyrolysis and particulate release. Consequently, the need for particulate control appears unnecessary.

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[21] Ibid Footnote 18

[22] Federal Register 40 CFR 264.343

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10.0 BUILDING HOT GAS DECONTAMINATION SYSTEM COST

Figures 10-1 and 10-2 present conceptual flow diagrams for hot gas decontamination systems for treating two different size building areas. System #1 (Figure 10-1) would be applicable for treatment of a concrete block or concrete wall building area of approximately 30 ft x 40 ft x 12 ft height and System #2 (Figure 10-2) for a building area of about 100 ft x 60 ft x 15 ft height. Larger buildings or multi-story buildings would be partitioned off with insulated steel sheet such that only one story or one area would be treated at a time. (Selected building area sizes were based on conversations with USATHAMA personnel.)

The two flow diagrams, one for each system, indicate the operating conditions during the initial and final heating of the building. Each of the two proposed systems incorporate a single burner for treating the building off-gases and supplying heated gas (air) to the building, thus conserving energy via the recycle stream. While it would be desirable (for explosion risk safety considerations) to use an inert hot gas atmosphere to heat the building area (by consuming most of the oxygen in the burner system), it is not possible to operate with an inert (oxygen-free) gas stream in this process because of air in-leakage to the building while operating under negative pressure. (There will always be some leakage through the thermal expansion joists between the steel sheets used to close-off the area being heated.) The estimated oxygen concentration in the gas streams from the treated areas is also shown in Figures 10-1 and 10-2. We have assumed that incineration for one second at 1800°F (our sampling conditions for the second pilot test) would produce an acceptable emission level from the afterburner.

The heating cycle for the building with the gas flow rates and temperatures shown in Figures 10-1 and 10-2 would be approximately 20 hours to obtain a 450 to 500°F (inside) wall temperature, followed by a four-hour hold at temperature (450 to 500°F). The estimated minimum and maximum heat inputs to the burner and building being treated would be as follows:

System	Building Size	Heat Input (10^6 Btu/hr) Burner	Building	Heat Cycle
#1	30 ft x 40 ft x 12 ft ht.	4.9	1.6	Initial Heatup (Minimum Input)
#1	30 ft x 40 ft x 12 ft ht.	7.4	2.7	Final Heatup Prior to Hold at Temp. (Maximum Input)
#2	60 ft x 100 ft x 15 ft ht.	23.2	7.0	Initial Heatup (Minimum Input)
#2	60 ft x 100 ft x 15 ft ht.	34.3	11.0	Final Heatup Prior to Hold at Temp. (Maximum Input)

10-1

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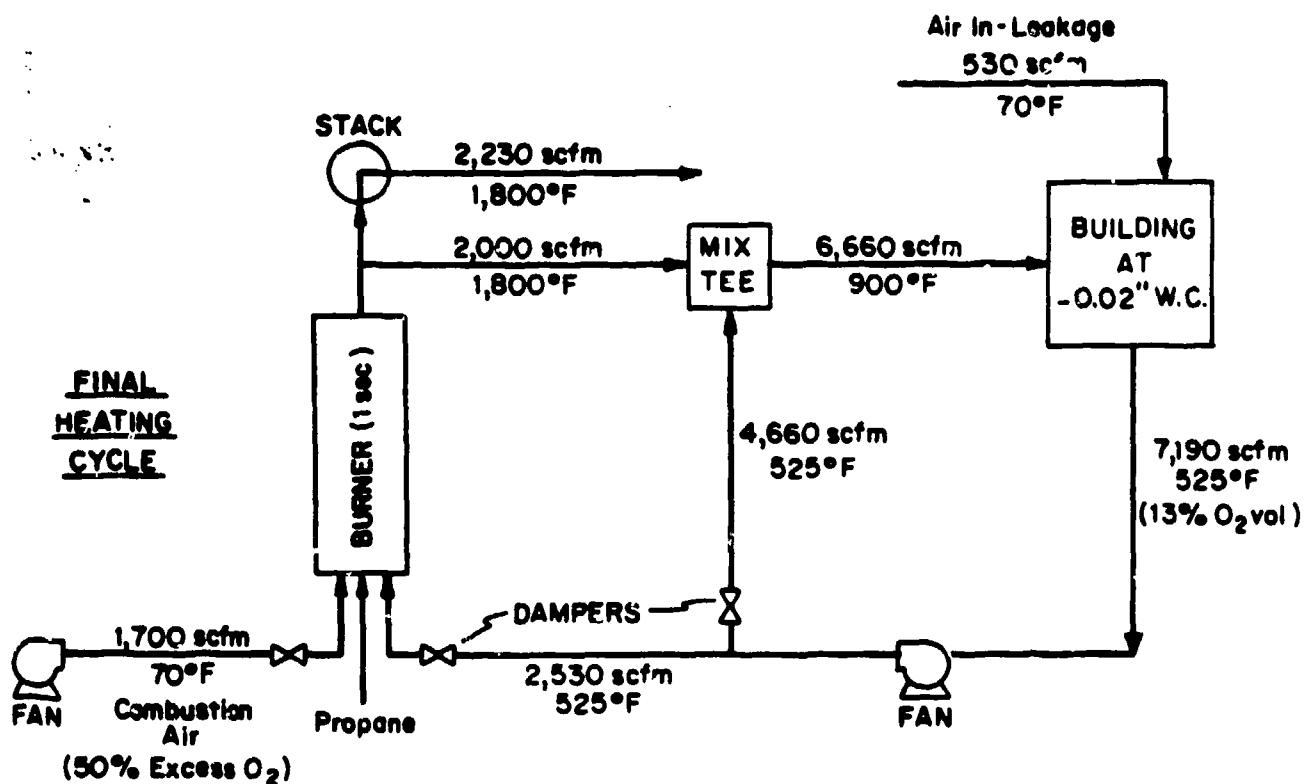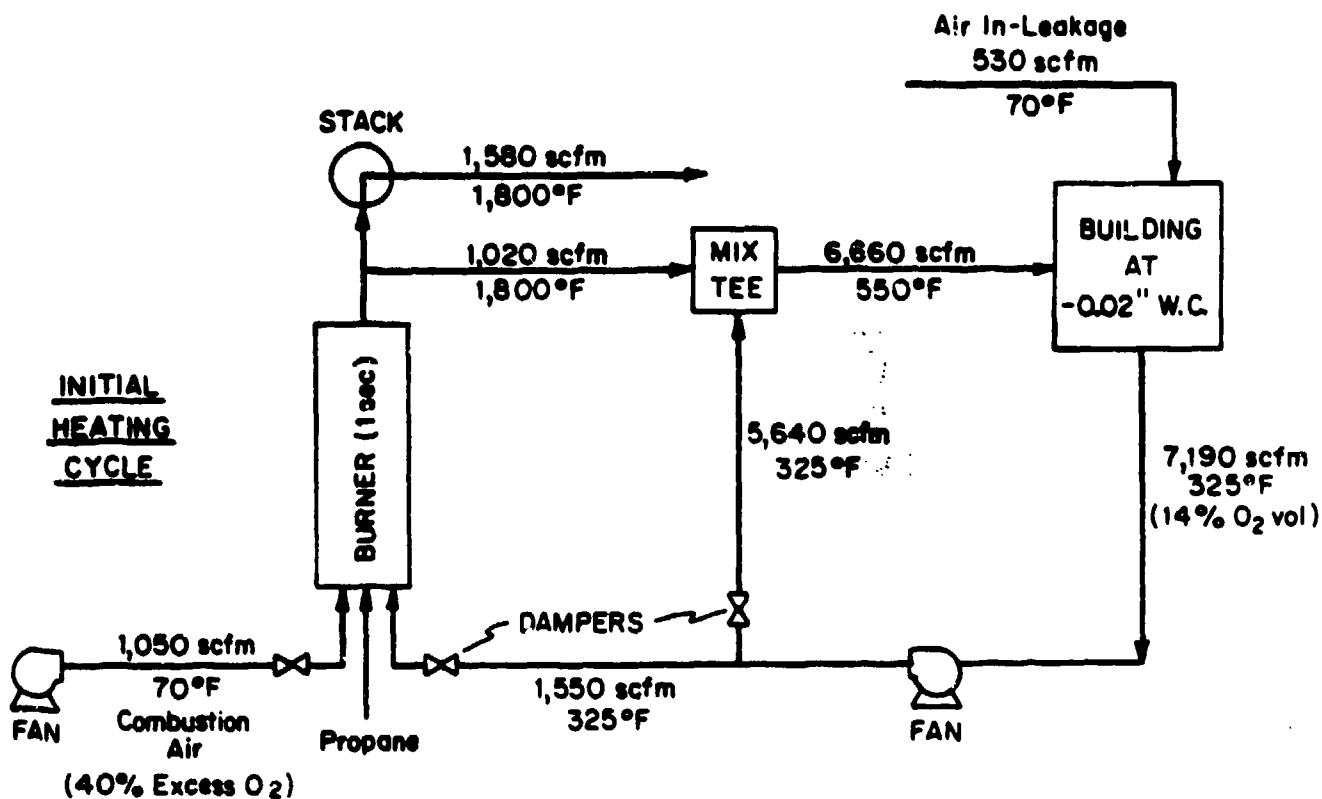
FIGURE 10-1 SMALL BUILDING AREA HOT GAS DECONTAMINATION SYSTEM
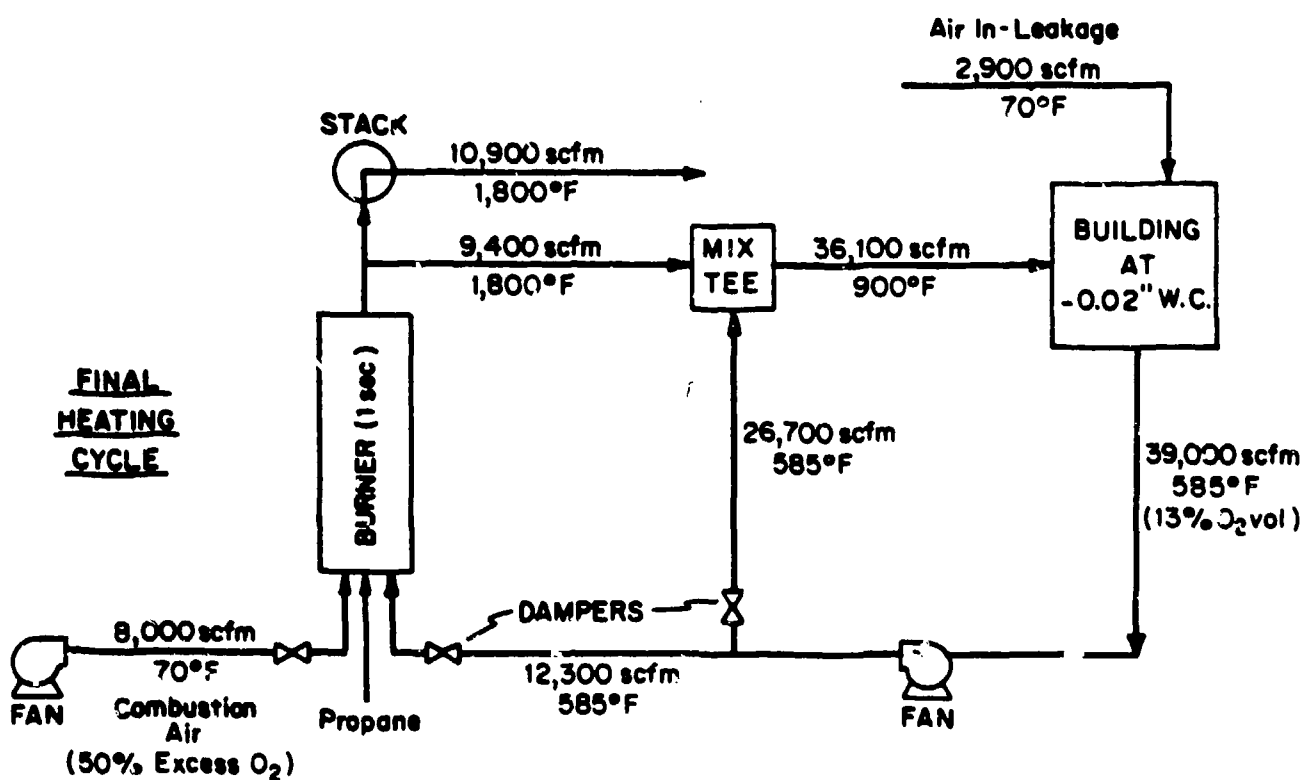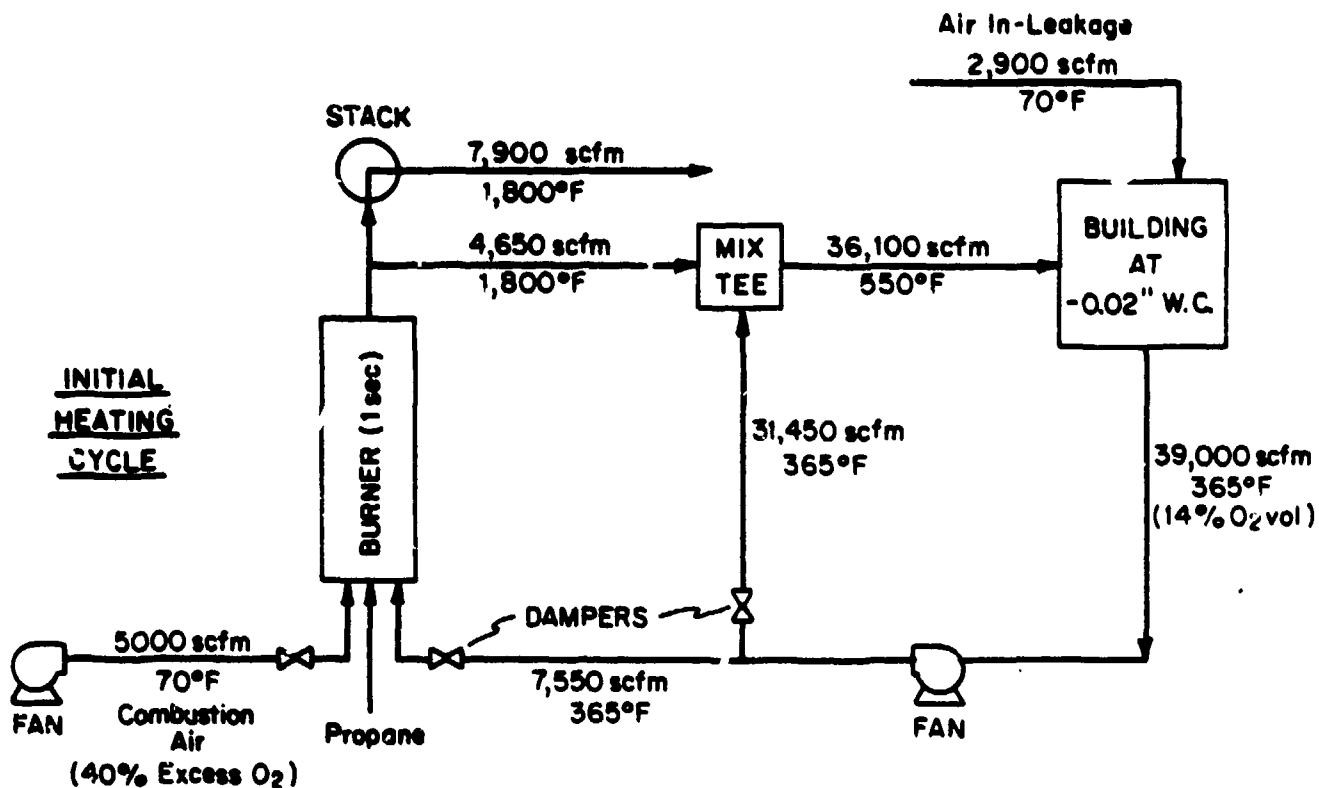
FIGURE 10-2 LARGE BUILDING AREA HOT GAS DECONTAMINATION SYSTEM

10-3

It is possible that the total (burner) heat input to each of the two systems could be reduced by about 12% by reducing the excess combustion air from 50% to 25%, or by about 25% if the oxygen in the air in-leakage were sufficient to support complete combustion. However, reducing the excess combustion air would make the system more difficult to control.

Heat recovery is also possible through use of a heat exchanger to recover heat from the afterburner stack gas. This could save up to 30% of the burner fuel requirements, but would require costly high temperature refractory materials for construction of the heat exchanger if an 1800°F operating temperature is necessary for adequate incineration of building off-gas.

The estimated capital and operating costs for the two burner systems is given in Table 10-1. This cost is for the burner system, gas handling equipment (fans, dampers, burner ductwork and stack) and controls. The cost of preparing the building for heating (e.g., removal of piping, wiring or wooden parts, partitioning, insulating and construction of ductwork, etc.) is based on the per square foot cost experienced at Cornhusker AAP, but this is very site specific and could vary considerably with other sites and types of building construction.

The estimated purchase cost of the burner system for the hot gas building decontamination process for the small building area (30 x 40 x 12 ft ht.) is about $150,000; while the estimated cost for the burner system for the larger building area (100 x 60 x 15 ft ht.) is about $210,000.

The burner systems would be operable by one person but, for safety reasons, additional personnel might be required (e.g., safety or fire fighting personnel). The cost for propane fuel for heating the two building areas (at $1 per gallon or $12 for 10^6 Btu) over a 24-hour period would be about $1800 for the smaller building area and $8200 for the larger building area.

The cost of electricity (mainly for the gas handling equipment) for 24-hour operation of the system (one decontamination run) would be about $90 (at 10 cents per kWh) for the smaller building area and about $380 for the larger building area.

In general, neither the capital cost of the burner system nor the operating costs (fuel, electricity and labor) for system operation seem particularly high. However, the costs associated with building preparation (equipment removal, room partitioning, insulation, duct work installation, etc.) prior to decontamination could be costly, especially due to the fact that these activities would be labor intensive. One important point to remember, though, is that the major benefit of this decontamination process is that it has the potential for rendering the building usable and available for excessing operations. If this does prove true, than the reuse potential of a building decontaminated in this fashion must be taken into account in any comparative cost analysis with other destructive-type decontamination processes.

10-4

TABLE 10-1

ESTIMATED CAPITAL/OPERATING COSTS FOR TRANSPORTABLE HOT GAS BUILDING DECONTAMINATION SYSTEMS

I. Capital Cost

Burner Size (10^6 Btu/hr)	Building Size (ft)	Capital Costs (1986, Thousands $)				
		Burner System with Controls, Fans & Dampers	Ductwork and 50 ft Stack (1 story bldg)	Equipment Installed[1] Cost	Building Modification[2] & Insulation	Total System Cost
7.5	30 x 40 x 12 ht.	95	58	175	50	225
35.0	60 x 100 x 12 ht.	131	78	245	200	445

II. Selected Operating Costs (per 24 hr Operation)

Burner Size	Propane ($1/gal)	Electricty (10¢/kWh)	Labor[3]	Gas Analysis	Explosive & Structural Sampling	Building Restoration
7.5 x 10^6 Btu/hr	1800	90	600	(4)	(5)	(5)
35.0 x 10^6 Btu/hr	8200	380	600	(4)	(5)	(5)

Notes:
(1) Includes installation cost (temporary burner foundations, wiring, plumbing), but not shipping cost.
(2) Based on test building at Cornhusker AAP, this cost would be very site specific.
(3) Six operators/24 hrs; one burner operator; one gas sampler operator/8 hr shift.
(4) Does not include fire protection or security personnel.
(5) Dependent on EPA requirements.
(5) Dependent on U.S. Government requirements.

Source: Arthur D. Little, Inc. and John Zink Co. Estimates

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11.0 CONCLUSIONS AND RECOMMENDATIONS

Overall, the use of the hot gas treatment appears to be a feasible means for decontaminating explosive contaminated buildings. More importantly, this hot gas decontamination process appears to be non-destructive (if performed within the proper temperature limits) and, therefore, could render treated buildings available for reuse or excessing operations.

A number of other conclusions can be drawn from these two pilot tests regarding: effect of heatup rate and final temperature on structural integrity; effect of temperature on decontamination efficiency; and off-gas emissions from this process. In addition, sufficient data was collected during these pilot tests such that a preliminary type design and cost estimate was prepared for a full-scale application of this process.

11.1 Effect of Heatup Rate and Temperature on Building Structural Integrity

The safety factor used in the design and construction of the actual buildings potentially being considered for decontamination may vary considerably, and a physical inspection or examination of construction drawings should be made before decontamination by this hot gas process is deemed appropriate. Such an inspection of the building in question would determine if the hot gas decontamination process has the potential to structurally weaken the building to below its safe design limit, or if additional structural support needs to be installed before or after decontamination.

The pilot plant tests we performed have shown that the loss in bend strength (tensile strength) is much greater after heating than is the loss in compressive strength. This must be taken into account when assessing the structural strength of the building for any structural members under shear or tensile load. However, for the most part, compressive strength is the more important of the two.

In contrast to the potential structural problems associated with heating building walls, heating the building floor to temperature may present a special problem if the floor is in contact with groundwater. In such a case, it may not be possible to bring the exterior floor surface up to the temperature necessary to achieve complete decontamination. This may not be a problem, however, if the floor is thick enough to effectively prevent migration of the contaminants back through the floor from the outside surface or if an additional sealing barrier (e.g., another layer of concrete) could be placed over the floor.

11.2 Recommended Heating Cycle

A rapid heatup rate, consistent with minimal structural damage, is desirable to minimize heat losses and overall heat input. Heat losses amount to about 80% of the heat transferred to the building as maximum temperature is approached, even with six inches of insulation around the walls and over the false ceiling. The heatup cycle used in the first pilot test, namely; inlet gas at 550°F for 4 hrs, then a 50°F increase per hour

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to 900°F, did not cause visible damage to the building, whereas the more rapid reheating (130 to 200°F/hr) and cooling of the test area in the second pilot test did cause visible structural damage. The recommended heating cycle is, therefore, based on the conditions of the first pilot test. The ultimate cause for fracture of the concrete materials is the difference in expansion rates of the aggregate and cement paste (which also undergoes shrinkage with water loss during the heating). Whether these differences are accentuated to a greater degree by the rapid heating or the thermal cycling is not known. Since multiple thermal cycling is not recommended in any case, the slower heating rate is also recommended to reduce potential for thermal shock and resultant structural damage.

In the recommended heating cycle, the initial hot gas temperature of 550°F would be maintained for four hours, followed by a 50°F increase per hour until the maximum inlet temperature was reached. (In this case, a building inlet gas temperature of only 900°F would be used as a means for limiting wall temperature.) The inlet gas temperature would then be gradually reduced, as required, to keep the inner wall surface at about 500°F until the exterior wall temperature reached about 400°F. Finally, the inside wall temperature would be maintained near this 500°F temperature long enough to keep the outside wall surface at a temperature of 400 to 450°F for a four-hour period.

11.3 Effect of Recommended Heating Cycle on Decontamination Efficiency

The recommended ultimate inner and outer wall temperatures to be achieved during the heating cycle are based on the following:

o Most of the explosive (TNT) contamination is on the inside surface of the wall;

o A 500°F inner surface temperature should reduce the TNT surface concentration to about 1 ug/sq cm;

o The interior residual explosive (TNT) concentration of a concrete block wall is reduced to about 0.11 ug/gm block at a temperature of 400°F or greater; and

o Limiting the overall wall temperature to between 400 to 500°F will minimize loss in concrete block wall structural strength (to about a 5% loss in compressive strength and 20% loss in tensile strength for non-reinforced concrete).

Although caustic spraying may have helped the surface decontamination of the blocks, it did not seem to be required for internal decontamination of the blocks. If a greater degree of surface decontamination is required than that provided by hot gas treatment alone at 500°F (a residual TNT level of less than 1 ug/sq cm block), the combination of hot gas and caustic spray might be considered as an alternative to increasing the hot gas temperature. Caustic spraying did not appear to weaken the blocks during heat treatment. (Spot caustic spraying in conjunction with hot gas treatment using visible and UV photography for locating explosive "hot

11-2

spots" might also be considered instead of general area saturation caustic spraying pretreatment.)

11.4 Building Off-Gas Destruction Efficiency

Destruction efficiency of the TNT (at 99.7% DRE) and the high boiling hydrocarbons (at about 97% DRE) is below the normal requirements specified for hazardous waste incineration, but concentrations of both leaving the afterburner are so low (ppb concentrations) that the 99.99% DRE may not be required. It appears likely that the thermal treatment of the contaminated off-gas from the heated building will be required, however, because of the potential for carryover of TNT and TNT decomposition products.

Provisions for particulate removal would seem to be unnecessary (based on our limited testing) in view of the very low particulate level measured in the afterburner exhaust (0.00014 gr/dscf).

11.5 Recommendations

The hot gas decontamination process for treating explosive contaminated buildings should be investigated further. Additional testing in the laboratory and pilot plant should include:

- o The effect of time/temperature on decontamination efficiency for TNT and other explosives on building materials.

- o The effect of the time/temperature conditions, required for adequate decontamination, on compressive strength of concrete block, solid concrete and reinforced concrete; and the effect of these conditions on the physical properties of the steel reinforcement (tensile strength, embrittlement, etc.).

- o The effect of time/temperature on decontamination efficiency of (steel) process equipment.

Such tests should be performed first on a laboratory-scale (for optimization of process conditions), followed by prove out using a pilot-scale system. In the pilot testing, building outlet and afterburner stack gas samples should be taken as the building wall temperature increases from 400 to 500°F (or above) to detect explosive emissions as well as paint breakdown products and insure that afterburner emission levels are measured during maximum organic input loadings.

When the issue of "safe" residual levels of explosives is resolved, the time/temperature decontamination efficiency and structural effects data should be reviewed. If the hot gas treatment alone is not efficient enough or results in too much structural damage, then the possibility of hot gas/spot caustic spraying should be investigated further.

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APPENDIX A

FIRST PILOT TEST - CHEMICAL ANALYSIS PROCEDURES AND RESULTS

FIRST PILOT TEST - CHEMICAL ANALYSIS PROCEDURES AND RESULTS

I. Introduction

A total of 114 field samples were received. The matrices consisted of the following:

- 2 MM5[1] XAD-2® sorbent traps
- 2 MM5 filters
- 1 MM5 condensate
- 1 Hydrant water
- 1 Milli-Q water
- 12 VOST[2] filters
- 12 VOST charcoal sorbent traps
- 12 VOST Tenax® sorbent traps
- 47 filter wipes (Whatman #42 Ashless 9 cm)
- 24 concrete block core samples

All samples were analyzed by High Performance Liquid Chromatography (HPLC) for 2,4,6-trinitrotoluene (TNT) and cyclotrimethylenetrinitramine (RDX). In addition, the MM5 sorbent samples and selected VOST sorbent samples were qualitatively analyzed by Gas Chromatography/Mass Spectrometry (GC/MS).

II. Analytical Method

A Waters Associates high performance liquid chromatography system was used to detect TNT, RDX, and 2,4-DNT. Specifically, the following components were included:

> Model 6000A Solvent Delivery System
> Model 440 Absorbance Detector
> Model 701B Intelligent Sample Processor (WISP)

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[1] MM5 - Modified Method 5 Sampling Train
[2] VOST - Volatile Organic Sampling Train

A-1

The conditions for the analytical method are outlined below:

Column:	Spherisorb ODS, 5 μm, 25 cm x 4.6 mm ID
Solvent System:	60% methanol, 5% acetonitrile, 35% water (by volume)
Detector:	UV at 254 nm
Flow Rate:	1.0 mL/min
Attenuation:	0.01 AUFS
Injection Volume:	15 ul
Integrator/Recorder:	Hewlett Packard 3390A, attenuation 2→2
Retention Time:	RDX - 5.1 minutes
	TNT - 6.3 minutes
	DNT - 8.0 minutes

III. Calibration Data

Calibration curves were established for RDX, TNT, and DNT throughout analysis. The complete range of calibration standards were analyzed prior to sample analysis. Standards were then interspersed between samples; one standard and four samples. The calibration curves were calculated using all standards analyzed. The concentration of standards, slope, intercept, correlation coefficient, and detection limit for each compound are listed in Table A-1. Figures A-1, A-2, and A-3 show plots of the calibration curves.

IV. Wipe Samples

A. Sample Preparation

Filter wipe samples were cut with a razor blade into quarters and placed in 2 oz. jars. Twenty milliliters of acetonitrile (CH₃CN) was added to the jars which were then covered with Teflon-lined caps. Samples were sonicated for 15 minutes. The first extraction was removed and filtered into two vials (analysis and storage). Remaining extract was discarded. The extraction was repeated a second time with an additional 20 mL of acetonitrile. Ten (10) mL of the second solvent extract was filtered, shaken, and transferred to sample vials.

The first and second extractions were analyzed separately to evaluate the completeness of the extraction process. Samples were diluted as necessary.

B. Sample Results

Sample results are presented in Table A-2. Results are shown as mass of TNT (mg) per 9 cm diameter filter.

C. Quality Control

Samples. Samples were desorbed in lots of 12. Each lot consisted of one blank filter and one spiked filter. Due to an error in spiking technique, five additional filters were spiked and anlyzed and the results reported.

A-2

TABLE A-1

LINEAR REGRESSION STATISTICS

	RDX	2,4,6-TNT	2,4-DNT
Standard Concentration - (ng/mL):	29.8	30	29.8
	59.7	60	59.7
	119	120	119
	199	200	199
	398	400	398
	995	--	--
Number of Standards Analyzed:	28	25	26
Slope [Height/(ng/mL)]:	0.0345	0.0711	0.0948
Intercept (Height):	-0.322	0.456	0.131
Correlation Coefficient:	0.9978	0.9973	0.9978
Limit of Detection - 2σ (ng/mL):	66.5*	40.6*	36.4*

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*Limit of Detection is calculated by the method of Hubaux
and Vos at the 95% confidence level.

Source: Arthur D. Little, Inc.
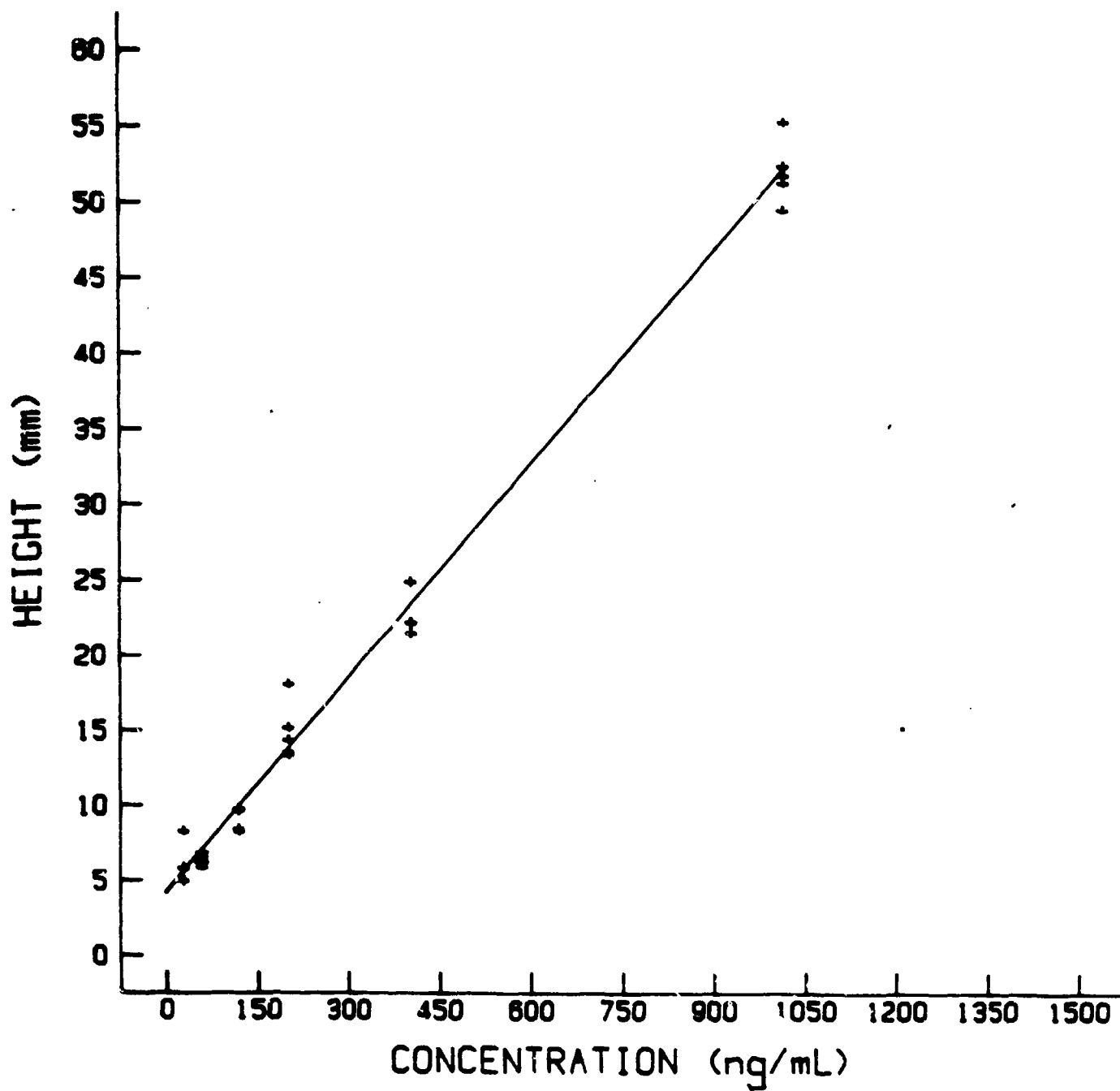
Arthur D. Little, Inc.

FIGURE A-1   TNT CALIBRATION CURVE

Source: Arthur D. Little, Inc.
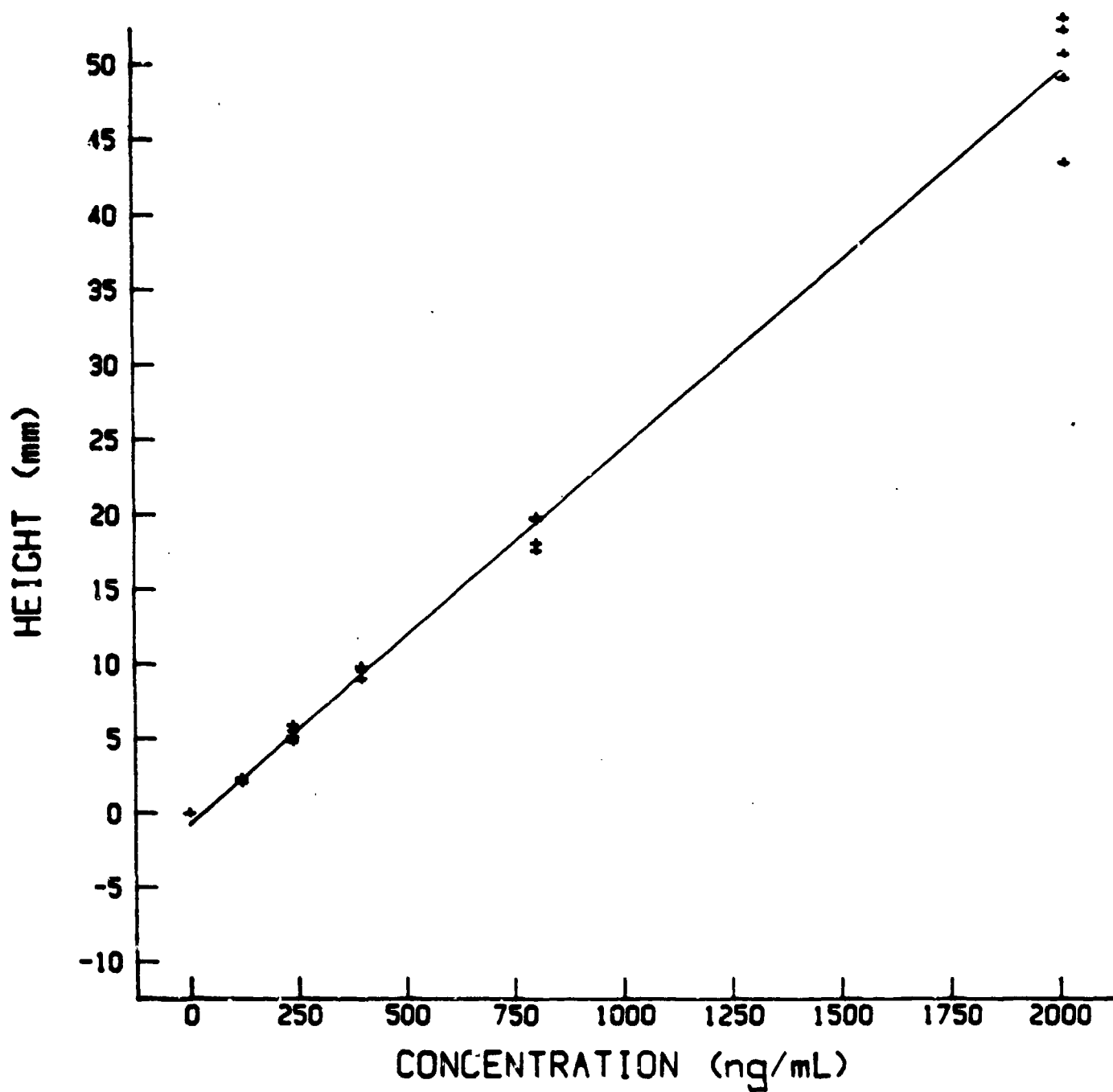
⚠ Arthur D. Little, Inc.

FIGURE A-2   RDX CALIBRATION CURVE

Source: Arthur D. Little, Inc.
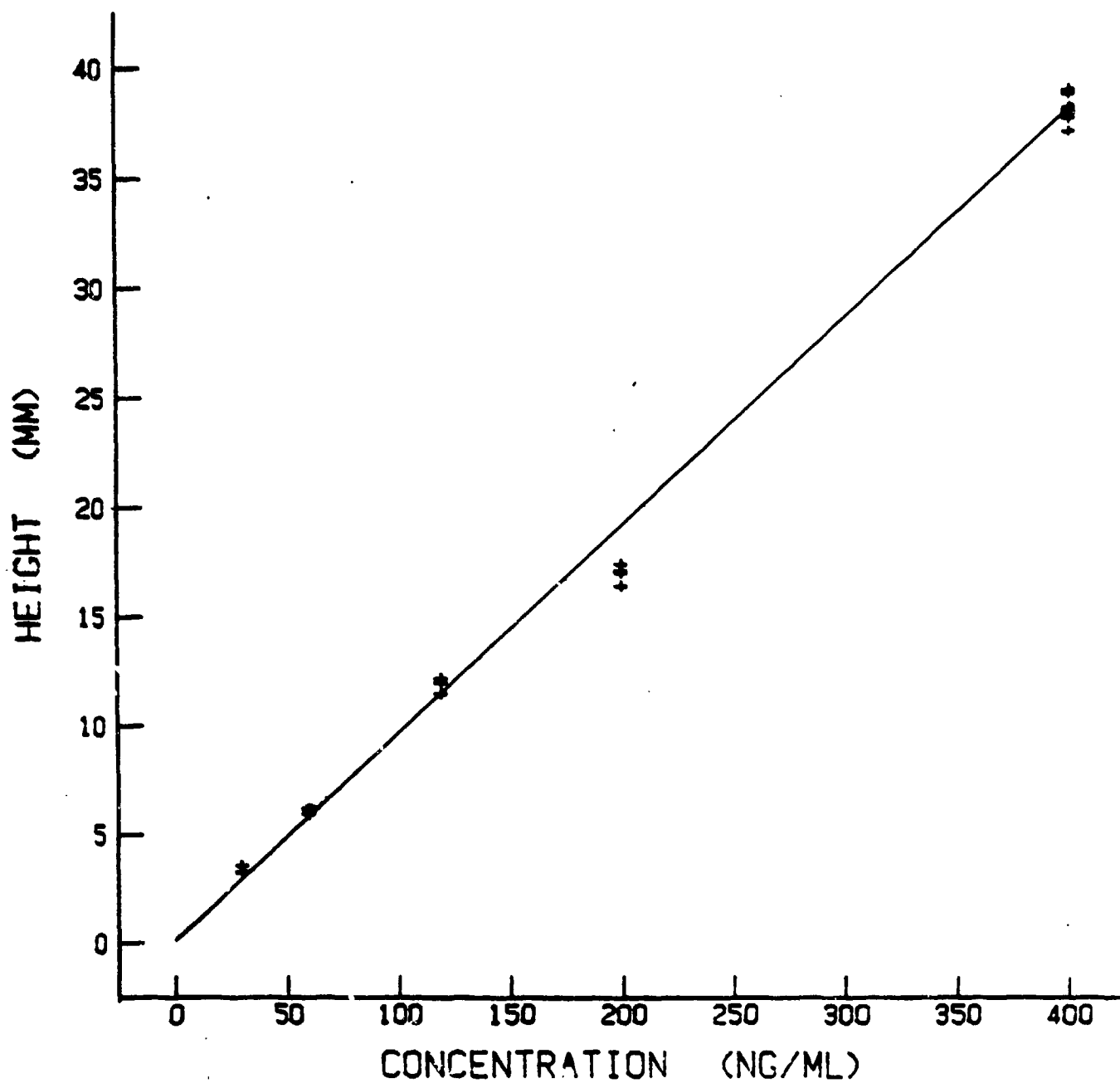
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FIGURE A-3   2,4-DNT CALIBRATION CURVE

Source: Arthur D. Little, Inc.

A-6

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TABLE A-2

WIPE SAMPLE RESULTS

Lab No.	Field No.	Mass TNT (mg)/Wipe
4457	9-7F	94.00
4458	9-7B	6.23
4459	9-8F	41.50
4460	9-8B	0.99
4461	9-9F	12.40
4462	9-9B	1.24
4463	9-12F	8.23
4464	9-12B	0.62
4465	9-13F	0.75
4466	9-13B	0.34
4467	9-14F	6.83
4468	9-14B	6.48
4469	9-17F	2.26
4470	9-17B	1.15
4471	9-19F	17.10
4472	9-19B	0.79
4473	9-27F	15.30
4474	9-27B	3.66
4475	9-28F	48.40
4476	9-28B	0.48
4477	9-29F	7.70
4478	9-29B	0.47
4479	9-30F	151.00
4480	9-30B	3.77
4481	AF	0.0021
4482	AB	0.0116
4483	BF	<0.00117
4484	BB	0.0013
4485	CF	<0.00117
4486	CB	<0.00117
4487	DF	<0.00117
4488	DB	0.0021
4489	EF	<0.00117
4490	EB	0.0013
4491	FF	<0.00117
4492	GF	<0.00117
4493	GB	<0.00117
4494	HF	<0.00117
4495	HB	<0.00117
4496	IF	<0.00117
4497	IB	0.0032
4498	JF	<0.00117
4499	JB	0.0065
4500	KF	<0.00117
4501	KB	0.01410
4502	LF	<0.00117
4503	LB	0.0197

Source: Arthur D. Little, Inc.

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TABLE A-3

WIPE MATRIX SPIKE AND RECOVERY DATA

Spike No.	Mass Spiked (ug TNT)	Mass Recovered (ug TNT)	% Recovery
5	24.4	29.7	122
6	24.4	29.9	122
7	24.4	29.1	119
8	24.4	29.2	120
9	24.4	30.1	123

Average Recovery:	121%
Standard Deviation:	1.64%
Relative Standard Deviation:	1.36%

Source: Arthur D. Little, Inc.

TABLE A-4

WIPE MATRIX REPLICATE DATA

Sample No.	Response 1 (ug TNT)	Response 2 (ug TNT)
4459A	39.40	36.70
4459B	3.48	3.36
4460A	0.86	0.81
4460B	0.15	0.16
4461B	0.87	0.84
4463A	7.62	7.36
4465A	0.66	0.67
4477A	6.97	7.01
4479A	156.00	144.00

Source: Arthur D. Little, Inc.

Arthur D. Little, Inc.

Blanks. Five blank filter samples were desorbed and analyzed. TNT was not detected.

Spikes. Five filters were spiked with 24.4 ug of TNT. This mass of TNT is approximately 20 times the Hubaux and Vos detection limit. Spikes were prepared and analyzed at this level to include the dilution step which was necessary for the filter samples collected prior to decontamination. Dilution for field samples were at first chosen based on color intensity (shade of red/pink) to avoid injecting a high concentration of TNT onto the chromatographic column. Therefore, quantifiction was for TNT alone (RDX was not detected in any of the diluted samples), and filters were spiked with TNT only. Table A-3 presents the extraction efficiency for the method.

Replicates. Nine samples were analyzed in replicate. Table A-4 contains the replicate response data.

V. Concrete Block Samples

A. Sample Preparation

Crushed concrete block samples were removed from plastic bags and transferred into tared 1-pint mason jars with Teflon®-lined lids and weighed. Acetonitrile (150 mL) was added to the jars and the contents mixed. The samples were agitated using sonication for 10 minutes with additional stirring after five minutes. Approximately 10 mL of the extracts were passed through 0.45 um Nylon-66 filters and the remainder decanted to a glass jar. A second extraction was completed as described above using an additional 100 mL of acetonitrile. Extracts were transferred into 4 mL autosampler vials and analyzed. The first and second extraction were analyzed separately to evaluate the completeness of the extraction process. Samples were diluted as necessary.

B. Sample Results

Sample results are given in Table A-5.

C. Quality Control

Samples. Samples were analyzed in lots of eight samples with two QC samples. QC samples were analyzed with each lot of samples.

Blanks. Two blank concrete block samples were extracted and analyzed. TNT was not detected.

Spikes. Six concrete block samples were spiked with TNT and extracted/analyzed. Spike levels were chosen to be representative of levels of TNT found in the field samples, with the exception of six samples which contain >497 ug of TNT. A spiking solution to spike at this level would have a concentration of 100 mg TNT/mL which is inappropriate. Recovery results are shown in Table A-6.

Replicates. Four samples were analyzed in replicate. Table A-7 lists replicate response data.

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TABLE A-5

CONCRETE BLOCK SAMPLES RESULTS

Lab No.	Field No.	Mass TNT (ug)	Sample Weight (grams)	Concentration (ug/g block)
4433	9-7	910.00	103.0	8.830
4434	9-8	8.73	110.5	0.079
4435	9-12	44.3	110.0	0.403
4436	9-14	1760.00	129.6	13.600
4437	9-19	76.00	110.2	0.689
4438	9-27	1370.00	116.8	11.800
4439	9-28	718.00	105.3	6.820
4440	9-30	336.00	106.1	3.170
4441	AF	19.80	151.5	0.131
4442	AB	497.00	150.3	3.310
4443	BF	14.30	208.9	0.068
4444	CF	22.00	207.6	0.106
4445	DF	23.70	174.9	0.135
4446	DB	39.80	120.1	0.331
4447	EF	9.21	132.6	0.069
4448	EB	20.80	86.0	0.242
4449	HF	17.60	152.7	0.116
4450	HB	10.60	138.1	0.077
4451	IF	<7.89	164.0	<0.048
4452	JF	9.21	147.8	0.062
4453	KF	24.60	94.8	0.260
4454	KB	692.00	67.5	10.200
4455	LF	<7.89	118.7	<0.066
4456	LB	210.00	129.8	1.620[1]

---

[1] See comment in text, Section V.A., Sample Preparation.

Source: Arthur D. Little, Inc.

Arthur D. Little, Inc.

TABLE A-6

CONCRETE BLOCK MATRIX SPIKE AND RECOVERY DATA

Spike No.	Mass Spiked (ug TNT)	Mass Recovered (ug TNT)	% Recovery	Average % Recovery
1	24.4	12.10	49.4	
2	24.4	9.53	39.0	45.9
3	24.4	12.00	49.2	
4	122.0	77.70	63.7	
5	122.0	104.00	85.1	70.5
6	122.0	76.40	62.7	

Source: Arthur D. Little, Inc.

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TABLE A-7

CONCRETE BLOCK MATRIX REPLICATE DATA

Sample No.	Response 1 (ug TNT)	Response 2 (ug TNT)
4433B	149	150
4436B	366	360
4438B	275	289
4440A	314	318

Source: Arthur D. Little, Inc.

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VI. VOST Samples

A. Sample Preparation

Tenax[*]. Sorbent was transferred from the stainless steel VOST trap into a 30 mL amber vial with Teflon[*]-lined cap. Twenty (20) mL of acetonitrile was added and the samples placed on the Burrell wrist action shaker for one hour. The extracts were filtered into autosampler vials and analyzed.

Charcoal. Sorbent was transferred from the stainless steel VOST trap into a 30 mL amber vial with Teflon[*]-lined cap. Ten (10) mL of acetonitrile was added and the samples placed on the Burrell wrist action shaker for one hour. The extracts were filtered into autosampler vials and analyzed.

Filter. Filter samples were placed in a 4 oz. jar with Teflon[*]-lined lid. Ten (10) mL of acetonitrile was added and the samples placed in an ultrasonic bath for 15 minutes. The extracts were filtered into autosampler vials and analyzed.

B. HPLC Sample Results

Samples were analyzed by HPLC for TNT and RDX. Results are given in Table A-8. Due to the recovery problems of DNT from the charcoal sorbent, results for these samples are in question. (Refer to VI.C.)

C. Quality Control

Blanks. A field blank VOST sample including charcoal sorbent, Tenax[*] sorbent, and filter was analyzed. RDX and TNT were not detected.

Spikes. 2,4-DNT was spiked into each charcoal and Tenax[*] sorbent sample as a surrogate. The average recovery of 2,4-DNT from the tenax was 90.4% (see Table A-9). However, the 2,4-DNT was not extracted from the charcoal sorbent. An alternative solvent for charcoal tube desorption will be evaluated prior to the next sampling trip.

D. GC Sample Results

Sorbent samples were analyzed by gas chromatography with a flame ionization detector (GC/FID) to determine Total Chromatographable Organics (TCO). The TCO value provides a quantitative measure of the amount of organic material in the sample which have boiling points between 100 and 300°C. Operating conditions for this analysis are given in Table A-10. A retention time window was established by analyzing C_7 (boiling point 98.4°C) and C_{17} (boiling point 302°C) as marker compounds.

Calibration standards were prepared at 5 concentration levels containing three hydrocarbons, i.e., octane (C_8), decane (C_{10}), and dodecane (C_{12}). A calibration curve was prepared plotting the concentration of the hydrocarbons versus their summed area. Linear regression statistics are given below:

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TABLE A-8

VOST SAMPLE RESULTS

Lab No.	Field No.	MASS (ug) RDX	MASS (ug) TNT	Volume Sampled (liters)	Concentration (ug/L) RDX	Concentration (ug/L) TNT
4397	1 Filter	< 0.66	< 0.41	42.8	< 0.015	< 0.011
4398	2 Filter	37.50	< 0.41	45.2	0.830	< 0.009
4399	3 Filter	< 0.66	< 0.41	51.7	< 0.013	< 0.008
4400	4 Filter	< 0.66	< 0.41	49.4	< 0.013	< 0.008
4401	5 Filter	< 0.66	< 0.41	49.8	< 0.013	< 0.008
4402	6 Filter	< 0.66	< 0.41	49.0	< 0.013	< 0.008
4403	7 Filter	< 0.66	< 0.41	28.6	< 0.023	< 0.014
4404	8 Filter	< 0.66	< 0.41	27.2	< 0.024	< 0.015
4405	9 Filter	< 0.66	< 0.41	27.7	< 0.024	< 0.015
4406	10 Filter	< 0.66	< 0.41	18.5	< 0.036	< 0.023
4407	11 Filter	< 0.66	< 0.41	15.1	< 0.044	< 0.027
4408	12 Filter	< 0.66	< 0.41	0.0		
4409	1 Tenax®	< 1.30	< 0.82	42.8	< 0.030	< 0.019
4410	2 Tenax®	< 1.30	< 0.82	45.2	< 0.029	< 0.018
4411	3 Tenax®	< 1.30	< 0.82	51.7	< 0.025	< 0.016
4412	4 Tenax®	< 1.30	< 0.82	49.4	< 0.026	< 0.017
4413	5 Tenax®	< 1.30	< 0.82	49.8	< 0.026	< 0.016
4414	6 Tenax®	< 1.30	< 0.82	49.0	< 0.026	< 0.017
4415	7 Tenax®	< 1.30	< 0.82	28.6	< 0.046	< 0.028
4416	8 Tenax®	< 1.30	< 0.82	27.2	< 0.048	< 0.030
4417	9 Tenax®	< 1.30	< 0.82	27.7	< 0.048	< 0.030
4418	10 Tenax®	< 1.30	< 0.82	18.5	< 0.072	< 0.044
4419	11 Tenax®	< 1.30	< 0.82	15.1	< 0.086	< 0.054
4420	12 Tenax®	< 1.30	< 0.82	0.0		
4421	1 Charcoal	< 0.66	< 0.41	42.8	< 0.015	< 0.011
4422	2 Charcoal	< 0.66	< 0.41	45.2	< 0.015	< 0.009
4423	3 Charcoal	< 0.66	< 0.41	51.7	< 0.013	< 0.008
4424	4 Charcoal	< 0.66	< 0.41	49.4	< 0.013	< 0.008
4425	5 Charcoal	< 0.66	< 0.41	49.8	< 0.013	< 0.008
4426	6 Charcoal	< 0.66	< 0.41	49.0	< 0.013	< 0.008
4427	7 Charcoal	< 0.66	< 0.43	28.6	< 0.023	< 0.014
4428	8 Charcoal	< 0.66	< 0.41	27.2	< 0.024	< 0.015
4429	9 Charcoal	< 0.66	< 0.41	27.7	< 0.024	< 0.015
4430	10 Charcoal	< 0.66	< 0.41	18.5	< 0.036	< 0.022
4431	11 Charcoal	< 0.66	< 0.41	15.1	< 0.044	< 0.027
4432	12 Charcoal	< 0.66	< 0.41	0.0	---	---

Source: Arthur D. Little, Inc.

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TABLE A-9

VOST SORBENT SPIKE AND RECOVERY DATA

Lab No.	Field No.	ug DNT Spiked	ug DNT Recovered	% Recovery
4409	1 Tenax	11.3	10.10	89.2
4410	2 Tenax	11.3	11.20	98.7
4411	3 Tenax	11.3	10.50	93.1
4412	4 Tenax	11.3	10.10	89.4
4413	5 Tenax	11.3	9.93	87.9
4414	6 Tenax	11.3	10.10	89.2
4415	7 Tenax	11.3	10.90	96.8
4416	8 Tenax	11.3	10.20	89.9
4417	9 Tenax	11.3	9.76	86.4
4418	10 Tenax	11.3	9.78	86.6
4419	11 Tenax	11.3	9.91	87.7
4420	12 Tenax	11.3	10.10	89.4
4421	1 Charcoal	11.3	< 0.36	0.0
4422	2 Charcoal	11.3	< 0.36	0.0
4423	3 Charcoal	11.3	< 0.36	0.0
4424	4 Charcoal	11.3	< 0.36	0.0
4425	5 Charcoal	11.3	< 0.36	0.0
4426	6 Charcoal	11.3	< 0.36	0.0
4427	7 Charcoal	11.3	< 0.36	0.0
4428	8 Charcoal	11.3	< 0.36	0.0
4429	9 Charcoal	11.3	< 0.36	0.0
4430	10 Charcoal	11.3	< 0.36	0.0
4431	11 Charcoal	11.3	< 0.36	0.0
4432	12 Charcoal	11.3	< 0.36	0.0

Source: Arthur D. Little, Inc.

TABLE A-10

GC/FID OPERATING CONDITIONS

Instrument:	Varian Vista 6000 with Flame Ionization Detector
Column:	Durabond-1 Fused Silica Capillary Column 0.32 mm ID, 30 meters, 0.25 um film thickness
Carrier Gas:	Helium at 2 mL/min
Makeup Gas:	Nitrogen at 28 mL/min
Column Temperature:	40°C (6 min.) $\xrightarrow{10°C/min}$ 300°C (10 min)
Injector Temperature:	200°C
Detector Temperature:	315°C
Range:	1×10^{-11}
Attenuation:	8

Retention Times:

C_7	- heptane	2.9 minutes
C_8	- octane	5.1 minutes
C_{10}	- decane	14.7 minutes
C_{12}	- dodecane	20.1 minutes
C_{17}	- heptadecane	21.4 minutes
DMF	- dimethyl formamide	2.5 minutes

Source: Arthur D. Little, Inc.

Arthur D. Little. Inc.

TABLE A-11

VOST TCO RESULTS

Lab No.	Field No.	Volume Sampled (liters)	Mass Hydrocarbon (ug)	Concentration Hydrocarbon (ug/L)
4409	1 Tenax	42.8	422	9.9
4410	2 Tenax	45.2	1020	22.6
4411	3 Tenax	51.7	1040	20.1
4412	4 Tenax	49.4	919	18.6
4413	5 Tenax	49.8	2200	44.2
4414	6 Tenax	49.0	780	15.9
4415	7 Tenax	28.6	942	32.9
4416	8 Tenax	27.2	885	32.5
4417	9 Tenax	27.7	2420	87.4
4418	10 Tenax	18.5	1890	102.0
4419	11 Tenax	15.1	< 414	< 27.4
4420	12 Tenax	0.0	< 414	---
4421	1 Charcoal	42.8	< 207	< 4.8
4422	2 Charcoal	45.2	211	4.7
4423	3 Charcoal	51.7	865	16.7
4424	4 Charcoal	49.4	839	17.0
4425	5 Charcoal	49.8	< 207	< 4.2
4426	6 Charcoal	49.0	991	20.2
4427	7 C' arcoal	28.6	913	31.9
4428	8 Charcoal	27.2	1210	44.5
4429	9 Charcoal	27.7	349	12.6
4430	10 Charcoal	18.5	258	13.9
4431	11 Charcoal	15.1	320	21.2
4432	12 Charcoal	0.0	< 207	---

Source: Arthur D. Little, Inc.

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```
Standard Concentration:        7.51
      (ug/mL)                  22.5
                               45.1
                               105
                               210
Number of Points:              7
Range (ug/mL):                 7.51-210
Slope [Area/(ug/mL)]:          1090
Intercept (Area):              6180
Correlation Coefficient:       0.9990
Limit of Detection - 2σ:       20.7 ug/mL
```

Samples were analyzed and the area between the retention times of C_7 and C_{17} summed. The TCO data was then reduced from the calibration curve. Sample results are shown in Table A-11.

A calibration standard of dimethylformamide at concentration 9.44 ug/mL was analyzed. The DMF peak eluted at 2.5 min. DMF was not detected in the VOST sorbent samples.

VII. Reanalysis of Concentrated VOST Samples by HPLC for TNT

A. Sample Preparation

Twelve VOST Tenax® sample extracts were concentrated to aid in the detection of TNT. A known volume of extract was pipetted into a calibrated conical tube. The tube was placed in a water bath at 45°C and nitrogen was passed over the extract. The concentration factor for each sample is given in Table A-12.

B. HPLC Analysis

HPLC conditions as described in previous analytical reports (dated 9/3/86 and 12/8/86) were used for the analysis of TNT.

C. Calibration Data

A calibration curve was established for TNT. The slope, intercept, correlation coefficient, and detection limit are shown below:

```
Concentration levels:          9
Number of points:              4
Range (ng/mL)                  117-976
Slope [Height/(ng/mL)]:        0.0154
Intercept (Height):            0.466
Correlation Coefficient:       0.9958
Limit of Detection 2σ (ng/mL): 153
```

D. Sample Results

HPLC sample results are also presented in Table A-12. Two samples (ADL 4411 and 4414) were difficult to quantify due to interfering peaks. The chromatogram for Sample 4411 contained a peak eluting at 6.82 minutes.

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TABLE A-12

HPLC RESULTS FOR CONCENTRATED VOST TENAX® SAMPLES

Lab No.	Field No.	Concentration Factor	Mass TNT (ug)	Volume Sampled (liters)	Concentration TNT (ug/L)
4409	1 Tenax ®	20	(0.14)[1]	42.8	0.0033
4410	2 Tenax ®	30	<0.10[2]	45.2	<0.0022
4411	3 Tenax ®	20	0.48	51.7	0.0093
4412	4 Tenax ®	20	<0.15	49.4	<0.0030
4413	5 Tenax ®	20	<0.15	49.8	<0.0030
4414	6 Tenax ®	30	interference	49.0	---
4415	7 Tenax ®	20	<0.15	28.6	<0.0052
4416	8 Tenax ®	30	<0.10	27.2	<0.0037
4417	9 Tenax ®	30	<0.10	27.7	<0.0036
4418	10 Tenax ®	20	<0.15	18.5	<0.0081
4419	11 Tenax ®	10	<0.31	15.1	<0.020
4420	12 Tenax ®	20	<0.15	0.0	---

[1]Value below Hubaux and Vos detection limit of 0.15 ug but greater than lowest standard.

[2]See text, HPLC Sample Results.

Source: Arthur D. Little, Inc.

There is a "shoulder" on the peak indicating a second component at the retention time of TNT. The quantitation for this sample shown in Table A-12 assumes the height of the "shoulder" is TNT alone.

The chromatogram for Sample 4414 has many offscale peaks between the retention times of 5.46 minutes to 6.60 minutes. These interfering peaks make the identification of the low levels of TNT impossible.

VIII. MM5 Samples

A.   Sample Preparation

XAD-2®. Sorbent was transferred to a glass thimble and placed in a soxhlet extractor. Sorbent was extracted with 400 mL of methylene chloride for 18 hours. The extracts were dried through sodium sulfate and concentrated using the Kuderna Danish apparatus to a volume of 10 mL. A 2 mL aliquot was used for HPLC analysis and a 3 mL aliquot was used for GC/MS analysis.

Filters. Tared filters were reweighed to determine weight of particulate collected. Results are presented in Table A-13. The filters were then transferred to glass thimbles and placed in a soxhlet extractor. Filters were extracted with 400 mL of methylene chloride for 18 hours. The extracts were dried through sodium sulfate and concentrated using the Kuderna Danish apparatus to a volume of 10 mL. A 2 mL aliquot was used for HPLC analysis.

Condensate/Water. The volume of samples was measured and then transferred to separatory funnels. Samples were acidified to a pH of 2 with 6N sulfuric acid and extracted three times with 30 mL of methylene chloride. Samples were then adjusted to a pH of 12 using 6N NaOH. Samples were extracted three times with 30 mL of methylene chloride, combining all extracts. The extracts were dried through sodium sulfate and concentrated using Kuderna Danish apparatus to a volume of 10 mL. A 2 mL aliquot was used for HPLC analysis.

B.   HPLC Sample Results

Samples were analyzed by HPLC for RDX and TNT. Results are given in Table A-14.

C.   "GRAV" Results

The gravimetric (GRAV) value provides a quantitative measure of the amount of organic material in the sample which have boiling points in excess of 300°C. One (1) mL of each sample extract was pipetted into a tared aluminum dish. After the solvent had evaporated, the dish was reweighed to determine residue weight. Sample results are also shown in Table A-13. Results are reported as mg of GRAV range organics per sample.

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TABLE A-13

"GRAV" DETERMINATION MM5 SAMPLES

Sample No.	Sample Description	Residue Weight
4390	XAD-2® trap	10.1 mg
4391	XAD-2® blank	0.5 mg
4392	Filter #62	0.0 mg
4393	Filter #63 (Blank)	0.3 mg
4394	Condensate	0.0 mg
4395	Hydrant water	0.0 mg
4396	Milli-Q water blank	0.0 mg

Sample No.	Sample Description	Particulate Weight
4392	Filter #62	1.5 mg
4393	Filter Blank #63	0.0 mg

Source: Arthur D. Little, Inc.

Arthur D. Little, Inc.

TABLE A-14

MODIFIED METHOD 5 SAMPLE RESULTS

		MASS (ug)		Volume Sampled (liters)	Concentration (ug/L)	
Lab No.	Sample Description	TNT	RDX		TNT	RDX
4390	XAD-2' Trap	4.99	<0.66	4550	0.0011	<1.4 x 10^{-4}
4391	XAD-2' Blank	<0.41	<0.66	0.0	---	---
4392	Filter #62	<0.41	<0.66	4550	<9.0 x 10^{-5}	<1.4 x 10^{-4}
4393	Filter #63 (Blank)	<0.41	<0.66	0.0	---	---
4394	Condensate	<0.41	<0.66	4550	<9.0 x 10^{-5}	<1.4 x 10^{-4}
4395	Hydrant Water	<0.41	<0.66	0.0	---	---
4396	Mill1-Q Water Blank	<0.41	<0.66	0.0	---	---

Source: Arthur D. Little, Inc.

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TABLE A-15

MM5 TCO RESULTS

Lab No.	Field Description	Volume Sampled (liters)	Mass Hydrocarbon (ug)	Corrected Mass Hydrocarbon (ug)	Concentration Hydrocarbon (ug/L)
4390	XAD-2® Trap	4550	3770	3249	0.714
4391	XAD-2® Blank	0.0	519	0.0	---
4392	Filter #62	4550	270	0.0	---
4393	Filter #63	0.0	512	0.0	---
4394	Condensate	4550	577	0.0	---
4395	Hydrant Water	---	591	0.0	---
4396	Milli-Q Water Blank	0.0	657	0.0	---
	Average Blank		521		

Source: Arthur D. Little, Inc.

⚠ Arthur D. Little, Inc.

D. GC Sample Results

TCO data was calculated following the procedure described for the VOST samples. These results are given in Table A-15. A contaminant in methylene chloride eluted at the same retention time as DMF, therefore, DMF was not determined in the MM5 samples.

IX. GC/MS Certification

Gas Chromatography/Mass Spectrometry (GC/MS) Class II Certification was achieved on July 23, 1986, according to the procedure described below. The instrument was calibrated using PFK, over a mass range of 35 to 350 amu. A 10 ppm solution of 2,4-Dinitrotoluene (DNT), 1,3,5-trinitrotoluene (TNT) and cyclotrimethylenetrinitramine (RDX) was analyzed by GC/MS. TNT and DNT were detected, however, RDX was not. One hundred ng (100 ppm) of RDX was detected, but chromatographed poorly. Therefore, since GC/MS is not the best method for analysis of RDX and RDX was not detected in previous analyses of concrete core and wipe samples, RDX was not included in certification.

Four standard sample blanks and four sample spikes containing 10 ppm TNT and DNT were analyzed on July 23, 1986. Each standard analysis was

alternated with a blank. All spikes were positive for detection of TNT and DNT by examination of the mass spectrum, library search, and retention time. All blanks were negative for these two analytes. The operating conditions of the GC/MS are shown in Table A-16.

X. GC/MS Sample Analysis

USATHAMA Cornhusker samples were analyzed by GC/MS following the procedures described for certification. The spectrometer was calibrated over a mass range of 35 to 350 amu. A blank and a 10 ppm standard of TNT and DNT were analyzed before and after the sample analyses. All samples were analyzed using a 1 ul injection volume of the sample and co-injecting 10 ng (0.5 ul) of an internal standard (D_{10} anthracene). The peak area of the internal standard was monitored in each run. Its retention time was 14 minutes, 47 seconds. Trinitrotoluene and dinitrotoluene were specifically looked for in each sample. The mass spectrum of every GC-eluting peak was manually examined and its spectrum searched against a library. Only sample 4390 (MM5 XAD-2 sample) was found to contain any components. The identities of these are given in attached Table A-17. None of the samples were found to contain TNT or DNT.

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TABLE A-16

GC/MS CONDITIONS

Instrument:	HP 5890 GC/VG70SEQ MS/DS
Column:	30 meter DB-1 fused silica capillary (0.32 mm ID, 0.25 um film thickness)
GC Temperature Program:	40°C (2 minutes) → 280°C at 12°C/minute
Injection Temperature:	240°C
Injection:	1 ul, splitless
GC/MS Transfer Line Temperature:	200°C
Retention Times:	DNT - 11 minutes, 49 seconds
	TNT - 13 minutes, 34 seconds
	RDX - 16 minutes
Mass Spectrometer Conditions:	Mass Range - 35-350
	Scan Speed - 0.5 sec/decade, 0.15 interscan delay

Source: Arthur D. Little, Inc.

TABLE A-17

COMPOUNDS IDENTIFIED IN SAMPLE 4390 (NM5 SORBENT)

Scan No.	Identification	Retention Time
5	Methylpentanol	2:04
10	Toluene	2:08
54	Phthalate	2:43
93	Silicone	3:13
128	Methylcyclopentanol	3:41
153	Dichlorocyclopentane	4:01
202	Benzaldehyde	4.39
277	Silicone	5:38
303	Silicone	5:59
324	1-phenylethanone	6:15
328	Dichlorocyclohexane	6:18
350	Methyl ester of hexadienoic acid	6:36
431	Ethylbenzaldehyde	7:40
446	Ethylbenzaldehyde (isomer)	7:51
457	Naphthalene	8:00
474	Silicone	8:13
487	Benzothiazole	8:24
504	Hydrocarbon	8:37
532	Ethylphenylethanone	8:59
550	Dimethylphenylethanone	9:13
600	Hydrocarbon	9:53
641	Silicone	10:25
672	Phenylene ethanone	10:49
690	Hydrocarbon	11:04
705	Silicone	11:15
720	Fluorinated/Chlorinated Hydrocarbon	11:27
727	Dimethyl Cyclohexadienedione	11:33
741	Hydrocarbon	11:44
746	Hydrocarbon	11:48
763	Silicone	12.01

COMPOUNDS IDENTIFIED IN SAMPLE 4390 (MM5 SORBENT)

Scan No.	Identification	Retention Time
775	Hydrocarbon	12:11
794	Silicone	12:26

811	N-(1,1-dimethylethyl)-4 methyl benzamide	12:39
818	Phthalate	12:44
842	Dimethylethyl, ethylpropanoic acid	13:03
858	Phenylmethyldimethylbenzene	13:16
902	Hydrocarbon	13:51
929	Fused ring aromatic - methyl substituted (MW236)	14:12
942	Silicone	14:22
951	Hydrocarbon	14:29
967	Phenanthrene	14:42
974	Internal Standard (D_{10}anthracene)	14:47
986	Hydrocarbon	14:57
1015	Hydrocarbon	15:20
1028	Hydrocarbon	15:30
1039	Hydrocarbon	15:39
1045	Phthalate	15:43
1085	Silicone	16:15
1130	Phthalate	16:20
1189	Chrysene	17:37
1252	Silicone	18:26
1482	Silicone/co-eluting with an unknown chlorinated hydrocarbon	21:27

Source: Arthur D. Little, Inc.

APPENDIX B

SECOND PILOT TEST - CHEMICAL ANALYSIS PROCEDURES AND RESULTS

SECOND PILOT TEST - CHEMICAL ANALYSIS PROCEDURES AND RESULTS

I. Introduction

A total of 67 field samples were received. The matrices consisted of the following:

    32 concrete block core samples

    18 filter wipe samples

    3 MM5[1] XAD-2® sorbent traps

    1 MM5 XAD-2® sorbent blank

    1 MM5 XAD-2® sorbent spiked blank

    3 MM5 filters

    1 MM5 filter blank

    3 MM5 condensate

    1 Milli-Q water

    3 MM5 methylene chloride rinses

    1 methylene chloride blank

All samples were analyzed by High Performance Liquid Chromatography (HPLC) for 2,4,6-trinitrotoluene (TNT), and cyclotrimethylenetrinitramine (RDX). MM5 samples were also analyzed by Gas Chromatography for Total Chromatographable Organics (TCO) and an aliquot of each extract was evaporated for gravimetric determination (GRAV).

In addition, MM5 inlet sorbent samples and sorbent blank were qualitatively analyzed by Gas Chromatography/Mass Spectrometry (GC/MS).

II. Analytical Method

A Waters Associates high performance liquid chromatography system was used to detect TNT and RDX. Specifically, the following components were included:

    Model 660 Solvent Programmer
    Model 6000A Solvent Delivery System
    Model 440 Absorbance Detector
    Model 701B Intelligent Sample Processor (WISP)

The conditions for the analytical method are outlined below:

---

[1]MM5 - Modified Method 5 Sampling Train

▲ Arthur D. Little, Inc.

Column:	Spherisorb ODS, 5 um, 25 cm x 4.6 mm ID
	(Supelco, Inc.)
Precolumn:	C-18 monofunctional, 5 um, 3 cm x 4.6 mm ID
	(Brownlee Labs)
Solvent System:	Initial Conditions:
	60% methanol, 5% acetonitrile, 35% water
	- hold 8 minutes
	- solvent program in 10 minutes
	Final conditions
	100% methanol
	- hold 10 minutes
Detector:	UV at 254 nm
Flow Rate:	1.0 mL/min
Attenuation:	0.01 AUFS, 0.1 AUFS, 0.2 AUFS
Injection Volume:	15 ul
Integrator/Recorder:	Hewlett Packard 3390A, attenuation 2↑2
Retention Time:	RDX - 4.6 minutes
	TNT - 5.8 minutes
	DNT - 7.2 minutes

III. Calibration Data

Calibration curves were established for RDX and TNT throughout analysis. The slope, intercept, correlation coefficient, and detection limit for each compound are listed in Table B-1. Figures B-1 and B-2 show plots of the calibration curves.

IV. Concrete Block Samples

A. Sample Preparation

Crushed concrete block samples (~ 130 grams) were removed from plastic bags and transferred into tared 1-pint mason jars with Teflon -lined lids and weighed. Acetonitrile (150 mL) was added to the jars and the contents mixed. The samples were agitated using sonication for 10 minutes with additional stirring after five minutes. Approximately 10 mL of the extracts were passed through 0.45 um Nylon-66 filters and the remainder decanted to a glass jar. A second extraction was completed as described above using an additional 100 mL of acetonitrile. Extracts were transferred into 4 mL autosampler vials and analyzed. The first and second extraction were analyzed separately to evaluate the completeness of the extraction process. The first extraction accounted for > 93% of the TNT recovered. Samples containing TNT at levels higher than the calibration range were reanalyzed using a less sensitive detector setting and more concentrated calibration standards.

B. Sample Results

Table B-2 presents the results of the analysis for TNT in concrete block core samples. The mass of TNT found in samples before and after decontamination is listed along with the concentration (ug TNT/gram concrete sample). The destruction efficiency has been calculated except in cases where TNT was not detected before decontamination.

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TABLE B-1

LINEAR REGRESSION STATISTICS

	RDX	2,4,6-TNT (low range)	2,4,6-TNT (high range)
Standard Concentration: (ng/mL)	120	30.3	1010
	240	60.6	2020
	400	121	5050
	800	202	10100
	2000	404	20200
		1010	40400
Number of Points:	22	29	10
Slope [Height/(ng/mL)]:	0.0252	.0474	18.1[c]
Intercept (Height):	-0.823	4.12	-1930[d]
Correlation Coefficient:	0.9955	0.9959	0.9998
Limit of Detection - 2σ (ng/mL)	252[a]	113[a]	1360[b]

---

[a] Detector Absorbance Units Full Scale = 0.01

[b] Detector Absorbance Units Full Scale = 0.2

[c] Slope [Area/(ng/mL)]

[d] Intercept (Area)

Source: Arthur D. Little, Inc.
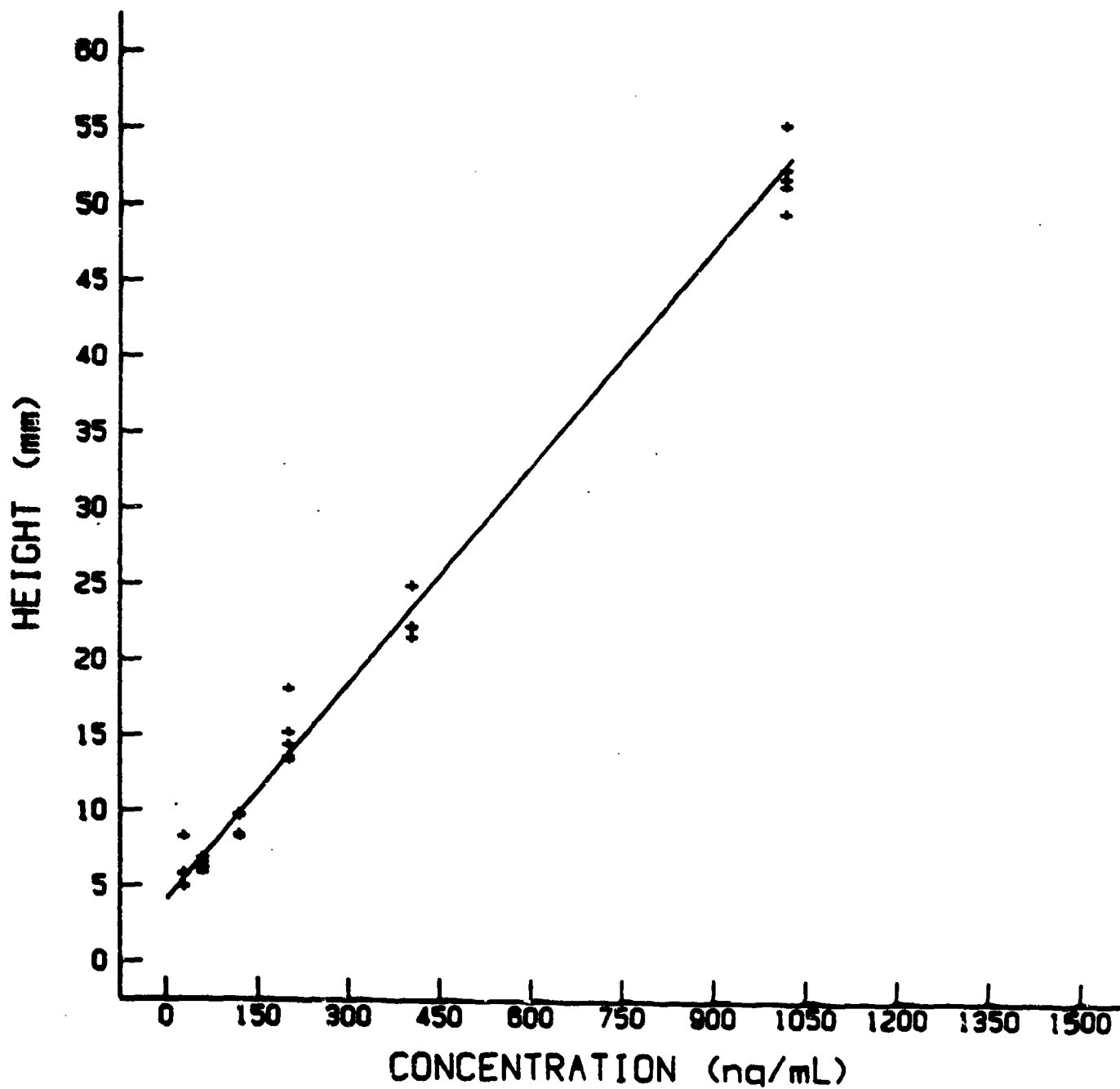
Arthur D. Little, Inc.

FIGURE B-1   TNT CALIBRATION CURVE

Source: Arthur D. Little, Inc.
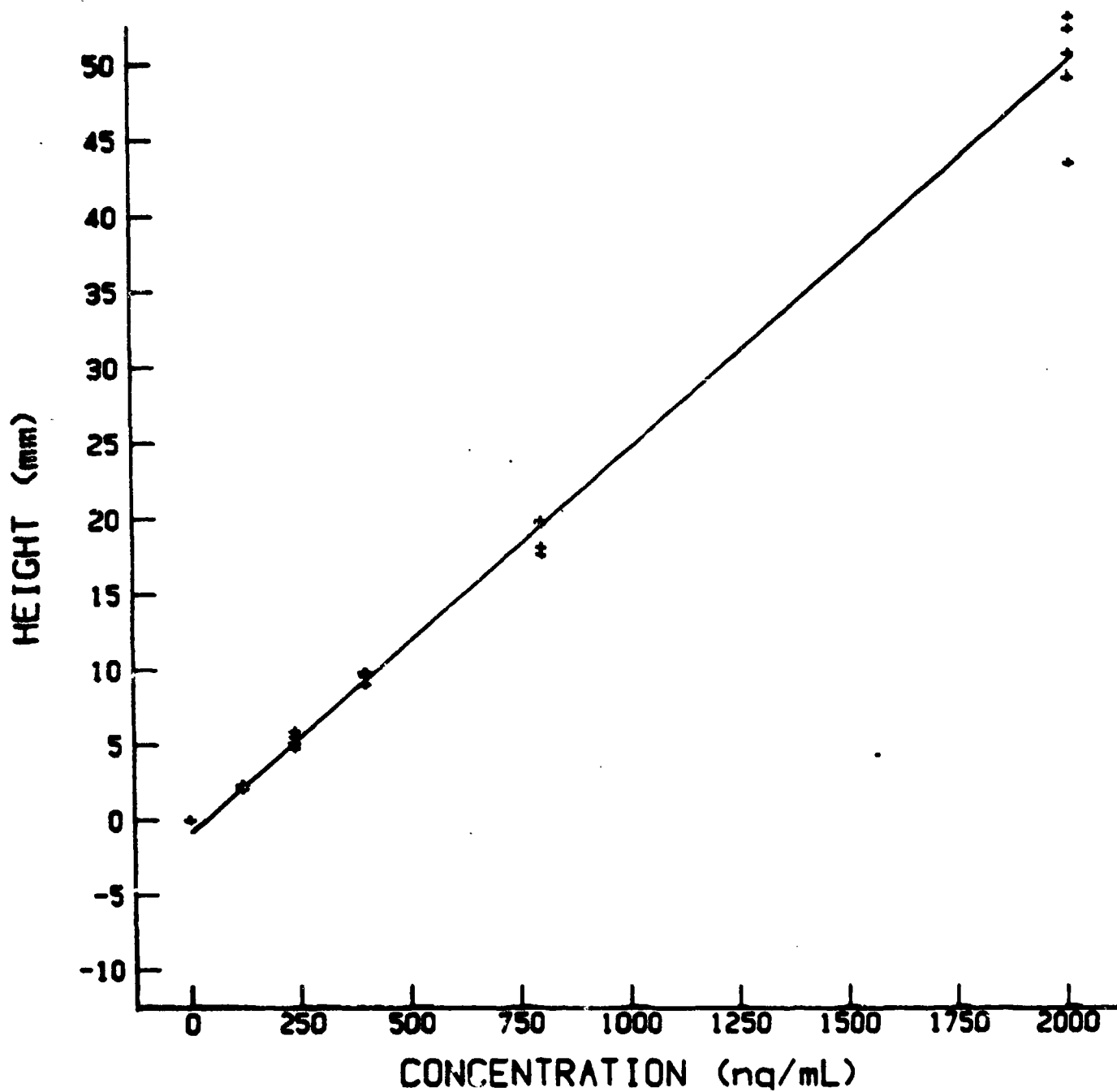
FIGURE B-2   RDX CALIBRATION CURVE

Source: Arthur D. Little, Inc.

TABLE B-2

TNT IN CONCRETE BLOCK SAMPLES

Concrete Block	Block Side	Before Decontamination			After Decontamination			Internal TNT Concentration (ug/gram)		Percent[a] Destruction Efficiency
		Sample Number	Sample Weight (grams)	ug TNT	Sample Number	Sample Weight (grams)	ug TNT	Before	After	
8-7	FRONT	4610	102.1	109	4626	126.0	<16.9	1.06	<.134	>87.4
7-30	FRONT	4611	102.5	<16.9	4627	123.2	<16.9	<0.16	<.138	-
7-2	FRONT	4612	116.8	1390	4628	117.3	<16.9	11.90	<.144	>98.8
7-20	FRONT	4613	114.8	186	4629	227.5	<16.9	1.62	<.074	>95.4
7-29	FRONT	4614	86.7	479	4630	105.7	<16.9	5.52	<.160	>97.1
7-8	FRONT	4615	96.7	<16.9	4631	147.4	lost	<.18	not analyzed	-
8-10A	FRONT	4616	122.6	683	4632	150.6	<16.9	5.57	<.112	>98.0
8-10B	FRONT	4617	101.9	1130	4633	165.8	<16.9	11.10	<.102	>99.1
8-27	BACK	4618	94.1	<16.9	4634	134.7	<16.9	<0.18	<.126	-
7-9	BACK	4619	114.3	2680	4635	156.2	<16.9	23.40	<.108	>99.5
7-2	BACK	4620	122.8	138	4636	122.1	<16.9	1.12	<.139	>87.6
7-20	BACK	4621	111.9	77.3	4637	238.4	<16.9	0.69	<.071	>89.7
7-29	BACK	4622	108.8	<16.9	4638	140.5	<16.9	<0.16	<.121	-
7-8	BACK	4623	112.3	259	4639	125.9	<16.9	2.31	<.135	>94.2
8-10A	BACK	4624	114.1	436	4640	149.3	<16.9	3.82	<.114	>97.0
8-10B	BACK	4625	96.1	17500	4641	163.6	<16.9	182.00	<.104	>99.9

[a] % Destruction Efficiency $= \left[\frac{(Z-Y)}{Z}\right] \times 100$

where: Z = concentration before decontamination (ug/g)
Y = concentration after decontamination (ug/g)

Source: Arthur D. Little, Inc.

The mass of TNT on samples before decontamination ranges from < 16.9 ug to 17.5 mg. For six concrete blocks, the front of the block and the back of the block were analyzed. In four cases, the front contained greater amounts of TNT than the back, but in two instances (Block 7-8 and 8-10B), the levels of TNT in the back portion were greater.

TNT was not detected in any sample after decontamination, therefore, destruction efficiency is dependent on the concentration of TNT prior to decontamination. Percent destruction efficiency ranges from > 87.4 to > 99.9.

C.   Quality Control

Blanks. A blank concrete block core sample was extracted and analyzed with each lot of field samples that were extracted. A total of three blank cores were analyzed; TNT and RDX were not detected. The detection limits for TNT and RDX are 16.9 and 37.6 ug/sample, respectively.

Spikes. Two cement block samples were spiked with TNT and three samples spiked with RDX. Spiking levels and recovery results are shown in Table B-3. The average recovery of TNT and RDX from concrete were 70.9% and 112%, respectively.

Replicates. Eight samples were analyzed in replicate. Table B-4 lists replicate response data. The average percent range for replicate injections was 6.9.

V.   Wipe Samples

A.   Sample Preparation

Filter wipe samples were cut with a razor blade into eighths and placed in 2 oz. jars. Twenty milliliters of acetonitrile (CH_3CN) was added to the jars which were then covered with teflon-lined caps. Samples were sonicated for 15 minutes. The first extraction was removed and filtered into two vials (analysis and storage). Remaining extract was discarded. The extraction was repeated a second time with an additional 20 mL of acetonitrile. Ten (10) mL of the second solvent extract was filtered, shaken, and transferred to sample vials. The first and second extractions were analyzed separately to evaluate the completeness of the extraction process. On average, the first extraction accounted for > 79% of the TNT recovered. Samples were reanalyzed at a less sensitive detector setting, as necessary.

B.   Sample Results

Sample results are presented in Table B-5. Results are shown as mass of TNT (ug) per 9 cm diameter filter before and after decontamination. RDX was not detected on any filter wipe sample.

Prior to decontamination, wipe samples taken from the front of a concrete block contained more TNT than samples taken from the back side. TNT ranged from 0.203 mg to 128 mg before decontamination.

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TABLE B-3

CONCRETE BLOCK MATRIX SPIKE AND RECOVERY DATA

Spike No.	Mass Spiked	Mass Recovered	% Recovery	Average % Recovery
1	120 μg TNT	92.4 μg TNT	77.0	70.9
3	120 μg TNT	77.8 μg TNT	64.8	
1	240 μg RDX	286 μg RDX	119.0	112.0
2	240 μg RDX	250 μg RDX	104.0	
3	240 μg RDX	271 μg RDX	113.0	

Source: Arthur D. Little, Inc.

⚠ Arthur D. Little, Inc.

TABLE B-4

CONCRETE BLOCK MATRIX REPLICATE DATA

Sample No.	Response 1 (μg TNT)	Response 2 (μg TNT)	Average (μg TNT)	% Range
4610A[a]	105	113	109	7.3
4612A	1200	1410	1300	16.0
4612B	83	86	85	4.2
4614A	439	518	479	16.0
4617A	1120	1120	1120	0.0
4619B	163	163	163	0.0
4620A	141	136	138	3.6
4621A	79	76	77	3.8

Average % Range 6.4

---

[a]"A" and "B" in the sample code refer to the first or second solvent extraction.

Source: Arthur D. Little, Inc.

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TABLE B-5

SUMMARY OF CONCRETE BLOCK SURFACE DECONTAMINATION RESULTS

Pilot Test 2 (October 1986)

SAMPLE NUMBER BEFORE	SAMPLE NUMBER AFTER	FILTER WIPE	CONCRETE BLOCK SIDE	SURFACE SAMPLE* TNT CONCENTRATION BEFORE	SURFACE SAMPLE* TNT CONCENTRATION AFTER	DESTRUCT. EFFIC. (%)	MAXIMUM BLOCK SUR- FACE TEMP. (°F)
4642	4651	8-7	FRONT	128000	3.10	>99.9	571
4643	4652	7-30	FRONT	4540	<2.17**	>99.9	582
4644	4653	7-8	FRONT	3580	3.75	>99.9	560
4645	4654	7-2	FRONT	38400	4.39	>99.9	587
4646	4655	7-2	BACK	2730	2.55	>99.9	480
4647	4656	7-20	BACK	200	<2.17	>98.9	513
4648	4657	8-10B	BACK	450	<2.17	>99.5	382
4649	4658	7-9	BACK	330	2.42	99.3	409
4650	4659	8-27	BACK	240	<2.17	>99.1	437

_____

* ug TNT per 9 cm filter paper wipe.
** Limit of detection.

Source: Arthur D. Little, Inc.

Arthur D. Little, Inc.

After decontamination, filter wipes contained less than 5 ug of TNT, giving a range of destruction efficiency > 99.1 to > 99.9%.

C.   Quality Control

Blanks. Two blank filter samples were desorbed and analyzed. TNT and RDX were not detected. The detection limits for TNT and RDX are 2.17 and 1.97 ug/sample, respectively.

Spikes. Three filter wipe samples were spiked with TNT and RDX. The mass of TNT spiked is at an intermediate level between the mass of TNT detected on "before decontamination" and "after decontamination" samples. Although RDX was not detected in any field samples, it was spiked onto blank filters to show solvent removal efficiency. Spiking levels and recovery results are shown in Table B-6. Average recovery of TNT and RDX from filter wipe samples was 111% and 114%, respectively.

Replicates. Ten samples were analyzed in replicate. Table B-7 lists replicate response data. The average percent range for replicate injections was 2.9.

VI.   **MM5 Samples**

A.   Sample Preparation

XAD-2®. Sorbent was transferred to a glass thimble and placed in a soxhlet extractor. Sorbent was extracted with 400 mL of methylene chloride for 18 hours. The extracts were dried through sodium sulfate and concentrated using the Kuderna Danish apparatus to a volume of 10 mL. A 2 mL aliquot of each extract was solvent exchanged into acetonitrile for HPLC analysis. A 3 mL aliquot was sent for GC/MS analysis.

Filters. Tared filters were reweighed in an attempt to determine weight of particulate collected. Adhesion of the glass fiber filter to the silicone O-ring prevented an accurate weight determination following the test. The filters were transferred to glass thimbles and placed in a soxhlet extractor. Filters were extracted with 400 mL of methylene chloride for 18 hours. The extracts were dried through sodium sulfate and concentrated using the Kuderna Danish apparatus to a volume of 10 mL. A 2 mL aliquot of each extract was solvent exchanged into acetonitrile for HPLC analysis.

Condensate Water/Methylene Chloride Rinse. The volume of samples was measured and then transferred to separatory funnels, combining the condensate water and methylene chloride rinse for each MM5 train. Samples were acidified to a pH of 2 with 6N sulfuric acid and extracted three times with 60 mL of methylene chloride. Samples were then adjusted to a pH of 12 using 6N NaOH. Samples were extracted three times with 60 mL of methylene chloride, combining all extracts. The extracts were dried through sodium sulfate and concentrated using Kuderna Danish apparatus to a volume of 10 mL. A 2 mL aliquot of each extract was solvent exchanged into acetonitrile for HPLC analysis.

⚛ Arthur D. Little, Inc.

TABLE B-6

FILTER WIPE MATRIX SPIKE AND RECOVERY DATA

Spike No.	Mass Spiked	Mass Recovered	% Recovery	Average % Recovery
1	25.0 μg TNT	27.4	109	
2	25.0 μg TNT	27.8	111	111
3	25.0 μg TNT	28.0	112	
1	28.0 μg RDX	32.4	116	
2	28.0 μg RDX	31.5	113	114
3	28.0 μg RDX	31.5	113	

Source: Arthur D. Little, Inc.

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TABLE B-7

FILTER WIPE MATRIX REPLICATE RESPONSE DATA

Sample No.	Response 1 (µg TNT)	Response 2 (µg TNT)	Average (µg TNT)	% Range
4642A[a]	118000	119000	118000	0.85
4642B	10100	10000	10000	1.00
4643A	4160	4120	4140	0.97
4644A	3120	3100	3110	0.64
4645A	35500	35300	35400	0.56
4645B	3010	3020	3020	0.33
4646A	2380	2390	2380	0.42
4647A	140	139	140	0.71
4647B	55.6	70.2	62.9	23.00
4650B	54.5	55.0	54.8	0.91

Average % Range 2.90

---

[a] "A" and "B" in the sample code refer to the first or second solvent extraction.

Source: Arthur D. Little, Inc.

⚠ Arthur D. Little Inc.

B.   HPLC Sample Results

Samples from the MM5 train were analyzed by HPLC for RDX and TNT. Results
for TNT are given in Table B-8. RDX was not detected. The detection
limit for RDX was 2.5 ug/sample or 0.42 ug/cubic meter.

In the first portion of the table, the mass of TNT (ug) and the
concentration (ug TNT/meter^3 gas sampled) for each sampling train is
given. The destruction efficiency has been calculated based on the
concentration in the Stack 1 and Inlet 1 MM5 train (sampled during the
same period of time).

The bottom portion of Table B-8 shows the mass of TNT collected on the
individual sampling matrices (XAD-2®, filter, condensate). For the two
inlet sampling trains, the majority (> 77%) of TNT was adsorbed on the
XAD-2® sorbent with the remaining being collected in the condensate. The
opposite was seen in the stack sampling train where TNT was detected only
in the condensate, perhaps due to the higher temperature of this MM5
train.

C.   Quality Control

Blanks. A field blank of each matrix type (XAD-2® sorbent, condensate,
and filter) was extracted and analyzed. TNT and RDX were not detected.
Blank results for TNT are also given in Table B-8. The detection limit
for RDX was 2.5 ug/sample.

Spikes. Four XAD-2® sorbent traps were spiked with TNT, extracted and
analyzed. Of these traps, one was spiked prior to the field trip and
brought out to Cornhusker AAP to show the stability of TNT on XAD-2®
sorbent. Spiking levels and recoveries are shown in Table B-9. The
average recovery of TNT from XAD-2® sorbent was 79.8%.

Replicates. Two samples were analyzed in replicate and one in triplicate.
This data is shown in Table B-9. The average percent range for replicate
injections was 2.8.

D.   "GRAV" Results

The gravimetric (GRAV) value provides a quantitative measure of the amount
of organic material in the sample which have boiling points in excess of
300°C. One (1) mL of each sample extract was pipetted into a tared
aluminum dish. After the solvent had evaporated, the dish was reweighed
to determine residue weight. Sample results are shown in Table B-10.
Results are reported as mg of GRAV range organics per sample.
The majority of organics with boiling point greater than 300°C were
collected on the sorbent, although the condensate extract accounts for >
20%. "GRAV" range organics were not collected on the filters, in fact,
the filter blank had a greater residue weight than any of the samples.
This may show inconsistencies between filters or inaccurate weight
determinations.

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TABLE B-8

CONCENTRATION OF TNT IN AFTERBURNER INLET AND OUTLET

MM5 Train (All Components)	Weight of TNT (ug)	Gas Volume (cu m)	Gas Conc. (ug/cu m)	Destruction[a] Efficiency (%)
Stack 1	38.4	5.5026	6.98	99.7
Inlet 1	14100	6.095	2310	----
Inlet 2	3230	6.226	519	----

****

MM5 Train	Matrix	Sample	TNT (ug)	Volume (cu m)	Conc. (ug/cu m)
Stack 1	XAD-2●	4587	<1.13	5.5026	<.205
Inlet 1	XAD-2●	4588	12300	6.095	2010
Inlet 2	XAD-2●	4589	3120	6.226	502
Field Blank	XAD-2●	4591	<1.13	5.9412[b]	<.190
Stack 1	Filter	4592	<1.13	5.5026	<.205
Inlet 1	Filter	4593	<1.13	6.095	<.185
Inlet 2	Filter	4594	<1.13	6.226	<.181
Field Blank	Filter	4595	<1.13	5.9412	<.190
Stack 1	Condensate	4596	38.4	5.5026	6.98
Inlet 1	Condensate	4597	1760	6.095	289
Inlet 2	Condensate	4598	113	6.226	18.1
Field Blank	Condensate	4599	<1.13	5.9412	<.190

[a] % Destruction Efficiency = $\left[\dfrac{Z - Y}{Z} \right] \times 100$ where Z = concentration in inlet 1
where Y = concentration in stack 1

[b] Average of three MM5 train volumes

Note: 1000 ug/cu m = 0.1 ppm (vol/vol) TNT

Source: Arthur D. Little, Inc.

TABLE B-9

QA/QC FOR MM5 SAMPLES

Spike and Recovery Data

Spike No.	Mass Spiked (ug TNT)	Mass Recovered (ug TNT)	% Recovery	Average % Recovery
4590*	10.1	8.85	87.6	
A	10.1	7.99	79.1	79.8
B	101	78.1	77.3	
C	101	76.0	75.3	

Replicate Data

Sample No.	Response 1 (ug TNT)	Response 2 (ug TNT)	Response 3 (ug TNT)	Average (ug/TNT)	% Range
4588	12300	12330	12130	12250	1.6
4589	3220	3030		3120	6.1
4597	3520	3540		3530	0.57

Average % Range   2.8

---

*Spiked at Arthur D. Little, Inc., then taken to Cornhusker AAP and returned to Arthur D. Little, Inc.

Source: Arthur D. Little, Inc.

⚠ Arthur D. Little, Inc.

TABLE B-10

HYDROCARBONS (bp > 300°C) IN EXHAUST SAMPLES

Sample No.	Sample Description	Residue Weight (mg)
4587	Stack 1 XAD-2	3.0
4588	Inlet 1 XAD-2	87.0
4589	Inlet 2 XAD-2	139.0
4591	Field Blank XAD-2	2.8
4592	Stack 1 Filter	0.5
4593	Inlet 1 Filter	1.5
4594	Inlet 2 Filter	2.0
4595	Field Blank Filter	2.5
4596	Stack 1 Condensate	3.0
4597	Inlet 1 Condensate	23.3
4598	Inlet 2 Condensate	24.0
4599	Field Blank Condensate	0.5

Source: Arthur D. Little, Inc.

E.   GC Sample Results

MS samples were analyzed by gas chromatography with a flame ionization detector (GC/FID) to determine Total Chromatographable Organics (TCO). The TCO value provides quantitative measure of the amount of organic material in the sample which have boiling points between 100 and 300°C. Operating conditions for this analysis are given in Table B-11. A retention time window was established by analyzing C_7 (boiling point 98.4°C) and C_{17} (boiling point 302°C) as marker compounds.

Calibration standards were prepared at 5 concentration levels containing three hydrocarbons, i.e., octane (C_8), dodecane (C_{12}), and hexadecane (C_{16}). A calibration curve was prepared plotting the concentration of the hydrocarbons versus their summed area. Linear regression statistics are given below:

Standard Concentration:	7.51
(ug/mL)	22.5
	45.1
	105
Number of Points:	8
Range (ug/mL):	7.51-105
Slope [Area/(ug/mL)]:	874
Intercept (Area):	348
Correlation Coefficient:	0.9993
Limit of Detection - 2σ:	0.070
(mg/sample)	

Samples were analyzed and the area between the retention times of C_7 and C_{17} summed. The TCO data was then reduced from the calibration curve. Samples results are shown in Table B-12.

Table B-12 is divided into two portions; the top is a summary of hydrocarbons collected in each of the sampling trains with the destruction efficiency calculated on the concentration in the Inlet 1 and Stack 1 trains. The bottom portion of the table outlines the collection devices for each train showing that organics were collected on the sorbent and in the condensate.

F.   GC/MS Certification

Gas Chromatography/Mass Spectrometry (GC/MS) Class II Certification was achieved on July 23, 1986, according to the procedure described below. The instrument was calibrated using PFK, over a mass range of 35 to 350 amu. A 10 ppm (ug/mL) solution of 2,4-Dinitrotoluene (DNT), 1,3,5-trinitrotoluene (TNT) and cyclotrimethylene trinitramine (RDX) was analyzed by GC/MS. TNT and DNT were detected, however, RDX was not. One hundred ng (100 ppm) of RDX was detected, but chromatographed poorly. Therefore, since GC/MS is not the best method for analysis of RDX and RDX was not detected in previous analyses of concrete core and wipe samples, RDX was not included in certification.

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TABLE B-11

GC/FID OPERATING CONDITIONS

Instrument: Varian Vista 6000 with Flame Ionization Detector

Column: Durabond-1 Fused Silica Capillary Column
0.32 mm ID, 30 meters, 0.25 um film thickness

Carrier Gas: Helium at 2 mL/min.

Makeup Gas: Nitrogen at 28 mL/min.

Column Temperature: $40°C$ (6 min.) $\xrightarrow{10°C/min.}$ $300°C$ (10 min.)

Injector Temperature: $200°C$

Detector Temperature: $315°C$

Range: 1×10^{-11}

Attenuation: 8

Retention Times:

C_7	- heptane	2.03 minutes
C_8	- octane	3.51 minutes
C_{12}	- dodecane	13.6 minutes
C_{16}	- hexadecane	18.9 minutes
C_{17}	- heptadecane	20.1 minutes .

Source: Arthur D. Little, Inc.

Arthur D. Little, Inc.

GC ANALYSES OF HYDROCARBONS (TCOs) IN MM5 SAMPLES OF GAS ENTERING AND LEAVING THE AFTERBURNER - PILOT TEST 2

MM5 Train (All Components)	Weight of Hydrocarbons (mg)	Gas Volume (cu m)	Gas Conc. (mg/cu m)	Destruction[a] Efficiency (%)
Stack 1	2.34	5.5026	0.425	92.2
Inlet 1	33.2	6.095	5.45	----
Inlet 2	39.3	6.226	6.31	----

****

MM5 Train	Matrix	Sample No.	Hydrocarbons (mg)	Volume (cu m)	Conc. (mg/cu m)
Stack 1	XAD-2	4587	1.85	5.5026	0.336
Inlet 1	XAD-2	4588	22.5	6.095	3.69
Inlet 2	XAD-2	4589	38.0	6.226	6.10
Field Blank	XAD-2	4591	0.116	5.9412[b]	.0195
Stack 1	Filter	4592	<.0703	5.5026	<.0128
Inlet 1	Filter	4593	<.0703	6.095	<.0115
Inlet 2	Filter	4594	<.0703	6.226	<.0113
Field Blank	Filter	4595	<.0703	5.9412	<.0118
Stack 1	Condensate	4596	0.494	5.5026	0.0898
Inlet 1	Condensate	4597	10.7	6.095	1.76
Inlet 2	Condensate	4598	1.3	6.226	0.209
Field Blank	Condensate	4599	<.0703	5.9412	<.0118

[a] % Destruction Efficiency $= \left[\dfrac{Z - Y}{Z}\right] \times 100$ where Z = concentration in inlet 1

where Y = concentration in stack 1

[b] Average of three MM5 train volumes

Source: Arthur D. Little, Inc.

Arthur D. Little, Inc.

Four solvent blanks and four solution spikes containing 10 ppm (ug/mL) TNT and DNT were analyzed on July 23, 1986. Each standard analysis was alternated with a blank. All spikes were positive for detection of TNT and DNT by examination of the mass spectrum, library search, and retention time. All blanks were negative for these two analytes. The operating conditions of the GC/MS are shown in Table B-13.

The Rank Sum Test has been applied to the certification results as shown in Table B-14. The sum of the ranks from the blanks does not exceed 10; therefore, the certification is acceptable.

To insure that sample preparation did not effect the integrity of sorbent sample extracts, two XAD-2 samples spiked with 101 ug TNT and one XAD-2 sample blank were extracted with 10 mL methylene chloride and analyzed by GC/MS on November 4, 1986. The concentration of the spiked sample extract was 10.1 ug/mL TNT and was positively identified by GC/MS in both samples. The analysis of the XAD-2 sample blank gave a negative response.

G.   GC/MS Sample Analysis

USATHAMA Cornhusker samples were analyzed by GC/MS following the procedures described for certification. The spectrometer was calibrated over a mass range of 35 to 350 amu. A blank and a 10 ppm standard of TNT and DNT were analyzed before and after the sample analyses. All samples were analyzed using a 1 ul injection volume of the sample and co-injecting 10 ng (0.5 ul) of an internal standard (D_{10} anthracene). The peak area of the internal standard was monitored in each run. Its retention time was 14 minutes, 6 seconds.

Samples 4588 and 4589 (MM5 XAD-2 - Inlet 1 and Inlet 2, respectively) were analyzed by full-scanning on the VG70SEQ. An XAD-2 field blank was also analyzed. Sample results were subjected to a full library search on all major components (>5 ng/ul). A list of compounds and their scan number found in the MM5 samples is included as Table B-15. The Total Ion Count (TIC) shows relative amounts for each component. Chromatograms of each of the samples are shown in Figures B-3, B-4, and B-5. Peaks are identified by scan numbers which correspond to those listed in Table B-15. Data has been archived on hard disk and is available for further reduction, if necessary.

Trinitrotoluene was identified in Samples 4588 and 4589 at the levels shown below. In addition, a concrete core sample submitted to GC/MS gave a positive response for TNT:

| | TNT Concentration | |
Sample	Extract Conc.	Gas or Block Conc.
Inlet 1 XAD-2 - 4588	>10,000 ug/10ml	>1640 ug/cu m
Inlet 2 XAD-2 - 4589	> 1,000 ug/10ml	> 164 ug/cu m
Block 7-2, front, before heating - 4612A	= 750 ug/150ml	= 6.4 ug/gm block

Arthur D. Little, Inc.

TABLE B-13

GC/MS Conditions

Instrument:	HP 5890 GC/VG70SEQ MS/DS
Column:	30 meter DB-1 fused silica capillary (0.32 mm ID, 0.25 um film thickness)
GC Temperature Program:	40°C (2 minutes) → 295°C at 12°C/minute (hold 10 minutes)
Injection Temperature:	250°C
Injection:	1 ul, splitless
GC/MS Transfer Line Temperature:	200°C
Retention Times:	DNT - 11 minutes, 21 seconds TNT - 12 minutes, 55 seconds RDX - 16 minutes
Mass Spectrometer Conditions:	Mass Range - 35-350 Scan Speed - 0.5 sec/decade, 0.15 interscan delay

Source: Arthur D. Little, Inc.

Arthur D. Little, Inc.

TABLE B-14

RANK SUM TEST

Standard Sample	Results[1]	Rank	Average Rank[2]
Blank Solvent	NN	1	2.5
Blank Solvent	NN	2	2.5
Blank Solvent	NN	3	2.5
Blank Solvent	NN	4	2.5
Standard Solution 10 ng on column	PP	5	6.5
Standard Solution 10 ng on column	PP	6	6.5
Standard Solution 10 ng on column	PP	7	6.5
Standard Solution 10 ng on column	PP	8	6.5

---

[1] NN = Negative
PP = Positive

[2] Average Rank for Negative Results = $\dfrac{1 + 2 + 3 + 4}{4}$ = 2.5

Average Rank for Positive Results = $\dfrac{5 + 6 + 7 + 8}{4}$ = 6.5

Sum of Average Ranks for Blanks = 2.5 + 2.5 + 2.5 + 2.5 = 10

The criterion for acceptability is that the sum of the average ranks for blanks be less than or equal to 10. Therefore, certification is acceptable.

Source: Arthur D. Little, Inc.

B-23

TABLE B-15

TENTATIVELY IDENTIFIED COMPOUNDS

Sample 4588 - Inlet 1:

Scan No.	Compound	Total Ion Count
21	Benzaldehyde	3.60×10^8
44	Hydrocarbon	1.69×10^8
74	Phenol	3.32×10^8
87	Hydrocarbon	1.25×10^8
91	Hydrocarbon	9.55×10^7
99	Silylated Material/Mixture	2.40×10^8
124	Silylated Material	1.12×10^9
137	Hydrocarbon	8.58×10^7
142	Phenyl Ethanone	3.24×10^8
150	Hydrocarbon	1.19×10^8
159	Hydrocarbon	1.12×10^8
188	Nonanone	2.68×10^8
209	Heptanoic Acid	3.97×10^8
219	Hydrocarbon-Branched	2.59×10^8
234	Silylated Material	1.33×10^8
240	Ethyl Benzaldehyde	8.74×10^7
248	Dimethyl Benzaldehyde	5.23×10^8
269	Ethyl Phenol	2.69×10^8
272	Naphthalene	1.52×10^8
277	Silylated Material	6.15×10^7
302	Benzoic Acid	7.12×10^8
320	Hydrocarbon	1.34×10^8
328	Dihydropentylfuranone	2.33×10^8
336	Methyldodecane	5.58×10^7
347	Ethyl Phenyl Ethanone	1.93×10^8
352	Tetrahydropyranone	8.87×10^7
365	Ethyl Phenyl Ethanone	1.19×10^8
371	Hydrocarbon	6.63×10^7

△ Arthur D. Little, Inc.

TABLE B-15 (continued)

TENTATIVELY IDENTIFIED COMPOUNDS

Scan	Compound	Total Ion Count
402	Isobenzofuranone	7.58×10^7
409	Hydrocarbon	3.27×10^8
415	Hydrocarbon	2.43×10^8
427	Dihydropentyl Furanone	2.30×10^8
443	Branched Acid	2.53×10^8
458	Silylated Material	6.90×10^8
470	Decanoic Acid	2.89×10^8
480	Decanone	5.05×10^7
484	Hydrocarbon	1.74×10^8
496	1,3-Dinitro Toluene (isomer)	1.97×10^8
503	Hydrocarbon	4.93×10^8
518	Silylated Material	1.53×10^8
526	Phenylhexanone	7.42×10^7
538	Dimethylcyclohexadienedione	1.34×10^8
548	Long Chain Acid	2.33×10^8
558	DNT (targeted compound)	6.20×10^8
562	Branched Hydrocarbon	6.04×10^7
566	Trimethylnaphthalene	7.24×10^7
573	3,5-Dinitrotoluene	1.81×10^8
586	Branched Hydrocarbon	7.10×10^8
605	Silylated Material	1.44×10^9
617	Dimethyl Ethyl Benzamide	7.24×10^8
624	Phthalate	5.12×10^8
630	Dodecanoic Acid	6.48×10^8
636	Branched Hydrocarbon	1.23×10^8
642	Branched Hydrocarbon	1.21×10^8
648	Long Chain Acid	2.82×10^8
655	Hydrocarbon	6.51×10^7
660	Cyclohexadecane	1.81×10^8

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TENTATIVELY IDENTIFIED COMPOUNDS

Scan	Compound	Total Ion Count
664	Hydrocarbon	9.46×10^8
680	TNT (targeted compound)	5.86×10^8
699	Branched Hydrocarbon	1.22×10^8
703	Branched Hydrocarbon	5.86×10^8
718	Hydrocarbon	1.68×10^8
736	Silylated Material	1.04×10^9
746	Branched Hydrocarbon	4.51×10^8
765	Phenanthrene	3.58×10^8
774	Coelution: Long Chain Acid/ Internal Standard	6.21×10^8
784	Hydrocarbon	6.51×10^7
798	Phthalate	8.37×10^7
809	Hydrocarbon	4.64×10^8
818	Hydrocarbon	4.21×10^8
826	Phthalate	5.10×10^8
849	Silylated Material	8.58×10^8
865	Methyl Phenanthrene	7.73×10^7
876	Hydrocarbon	4.27×10^8
887	Phthalate	6.51×10^8
907	Long Chain Acid	6.37×10^8
917	Phenyl-bicyclohexyl	7.82×10^7
934	Long Chain Ester	6.21×10^7
951	Silylated Material	7.33×10^8
966	Tetrahydro Indacene Dione	9.50×10^7
978	Pyrene	3.64×10^8
990	Hydrocarbon	7.98×10^7

TABLE B-15 (continued)

TENTATIVELY IDENTIFIED COMPOUNDS

Sample 4589 - Inlet 2:

Scan	Compound	Total Ion Count
21	Benzaldehyde	7.78×10^8
61	Branched Hydrocarbon	3.08×10^8
74	Phenol	9.59×10^8
89	Hydrocarbon	3.01×10^8
104	Pyranone - saturated	7.37×10^8
113	Branched Hydrocarbon/Cumene	6.74×10^8
117	Hydroxy Benzaldehyde	5.26×10^8
125	Silylated Compound	1.26×10^9
143	Phenyl Ethanone	6.28×10^8
174	Hydrocarbon-unsaturated	3.41×10^8
183	Benzoic acid-methyl ester	1.86×10^8
189	Nonanone	3.53×10^8
211	Nitro-phenol	6.73×10^8
220	Hydrocarbon	3.53×10^8
244	Silylated Material	3.91×10^8
249	Ethyl Benzaldehyde	4.32×10^8
255	Ethyl Pyranone	9.82×10^7
264	Dimethyl Benzaldehyde	1.90×10^8
270	Ethyl Phenol	3.52×10^8
274	Naphthalene	1.94×10^8
279	Nonanol	2.40×10^8
283-290	Benzoic Acid	4.61×10^8
295	Silylated Material	6.39×10^8
349	Ethanone-Ethylphenyl	2.47×10^8
363	Isobenzofurandione?	5.48×10^8
367	Benzenedicarboxylic Acid	7.91×10^8
392	Decanoic Acid?	6.38×10^8

B-27

TENTATIVELY IDENTIFIED COMPOUNDS

Scan	Compound	Total Ion Count
403	Alcohol-long chain	1.68×10^8
408	Benzenedicarboxaldehyde	5.25×10^8
416	Hydrocarbon	2.92×10^8
431	Hydrocarbon	2.39×10^8
441	Benzopyranone	1.14×10^8
455	Benzoic Acid Ester	8.21×10^8
459	Benzofuranone	7.88×10^8
464-467	Benzoic Acid Ester	1.48×10^8
473	Benzenemethanol-Dimethyl	5.46×10^8
478	Indole-dione???	5.48×10^8
484	Benzoic Acid Ester	3.23×10^8
488	Ethanone-Phenylene (bis)	3.57×10^8
493	Hydrocarbon	2.02×10^8
504	Hydrocarbon	3.93×10^8
512	Indole-dione??	6.34×10^8
520	Indole-dione??	6.68×10^8
523	Indole-dione??	6.78×10^8
552	Undecanoic Acid	4.48×10^8
578	Branched Hydrocarbon	2.62×10^8
587	Nitrophenol	8.40×10^8
606	Silylated Material	1.13×10^9
627	Phthalate	2.80×10^8
630	Long Chain Fatty Acid??	6.11×10^8
665	Hydrocarbon	6.14×10^8
684	TNT	9.00×10^8
692	Hydrocarbon	1.54×10^8
704	Hydrocarbon	5.12×10^8

TABLE B-15 (continued)

TENTATIVELY IDENTIFIED COMPOUNDS

Scan	Compound	Total Ion Count
711	Hydrocarbon Unsaturated	9.54×10^7
724	Pentadecanone	2.81×10^8
728	Benzoic Acid Ester	6.98×10^7
732	Silylated Material	9.29×10^7
737	Silylated Material	1.07×10^8
747	C_{19} Hydrocarbon-Branched	3.58×10^8
764	$C_{14}H_{12}$ Aromatic	2.22×10^8
769	D_{10} Anthracene I.S.	1.57×10^8
775	Tetradecanoic Acid?	6.20×10^8
797	Phthalate	1.86×10^8
810	C_{18} Hydrocarbon-straight	3.27×10^8
819	Hydrocarbon (+) Phenylnaphthalene	2.87×10^8
827	Phthalate	4.49×10^8
850	Silylated Material	7.53×10^8
866	Long Chain Ketone	1.40×10^8
873	Methanone, Diphenyl Oxime?	1.50×10^8
877	Hydrocarbon	2.30×10^8
888	Phthalate	2.45×10^8
894	Phenyl-naphthalene	7.18×10^7
908	Long Chain Ester?	6.71×10^8
942	Hydrocarbon	1.40×10^8
948	Pyrene	9.63×10^7
953	Silylated Material	1.32×10^8
966	Phenanthrene-partially saturated	1.07×10^8
974	Hydrocarbon	7.07×10^7
979	Hydrocarbon	3.38×10^8
1031	Octadecanoic Acid?	1.72×10^8

TABLE B-15 (continued)

TENTATIVELY IDENTIFIED COMPOUNDS

Scan	Compound	Total Ion Count
1046	Silylated Material	6.90×10^8
1062	Hydrocarbon	8.83×10^7
1066	Hexadecanoic Acid-Hydroxyl Ethyl Ester	1.47×10^8
1086	Hydrocarbon	6.60×10^7
1103	Hydrocarbon	1.18×10^8
1119	Hydrocarbon	1.28×10^8
1132	Silylated Material	6.90×10^8
1212	Silylated Material	5.14×10^8
1227	Phthalate	9.53×10^7
1286,1355,1421	Silylated Material	3.38×10^8

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TABLE B-15 (continued)

TENTATIVELY IDENTIFIED COMPOUNDS

Sample 4591 - Field Blank:

Scan	Compound	Total Ion Count
16	Trichloropropene	5.38×10^6
23	Benzaldehyde	1.02×10^7
49	Pentachloroethane	1.02×10^7
110	Decane	7.45×10^6
145	Dichlorocyclohexane	1.96×10^7
168	Hexadienoic Acid, Methyl Ester	1.13×10^7
248	Ethyl Benzaldehyde	8.21×10^6
273	Naphthalene	9.53×10^6
348	Ethylphenylethanone	4.81×10^6
420	Cyclohexenylbenzene	2.67×10^7
442	Ester of Branched Chain Acid	1.26×10^7
459	Ester of Branched Chain Acid	1.68×10^7
556	Dichloro Methyl Phenol	7.01×10^6
570	Silylated Material	1.17×10^7
586	Branched Hydrocarbon	9.54×10^6
624	Phthalate	1.90×10^7
647	Ester of Branched Chain Acid	8.10×10^6
679	Propanediyl Benzene	6.49×10^6
768	Internal Standard	1.02×10^8
886	Phthalate	1.40×10^7
1110	Phthalate	1.03×10^7
1207	Silylated Material	5.64×10^6

Source: Arthur D. Little, Inc.
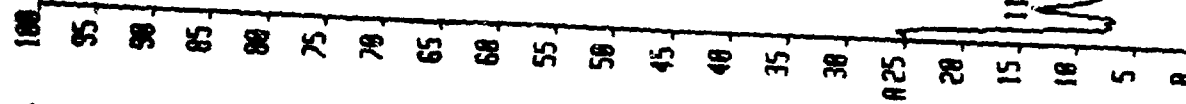
△ Arthur D. Little. Inc.

FIGURE B-3   GC/MS CHROMATOGRAM: SAMPLE 4588   INLET 1   XAD-2®

FIGURE B-3   GC/MS CHROMATOGRAM: SAMPLE 4588   INLET 1   XAD-2®(Continued)

Source: Arthur D. Little, Inc.

FIGURE B-4   GC/MS CHROMATOGRAM: SAMPLE 4589 INLET 2 XAD-2®

FIGURE B-4   GC/MS CHROMATOGRAM: SAMPLE 4589 INLET 2 XAD-2® (Continued)

Scan Number	800	1000	1200	1400
Time (min.)	14:36	17:07	19:45	22:22
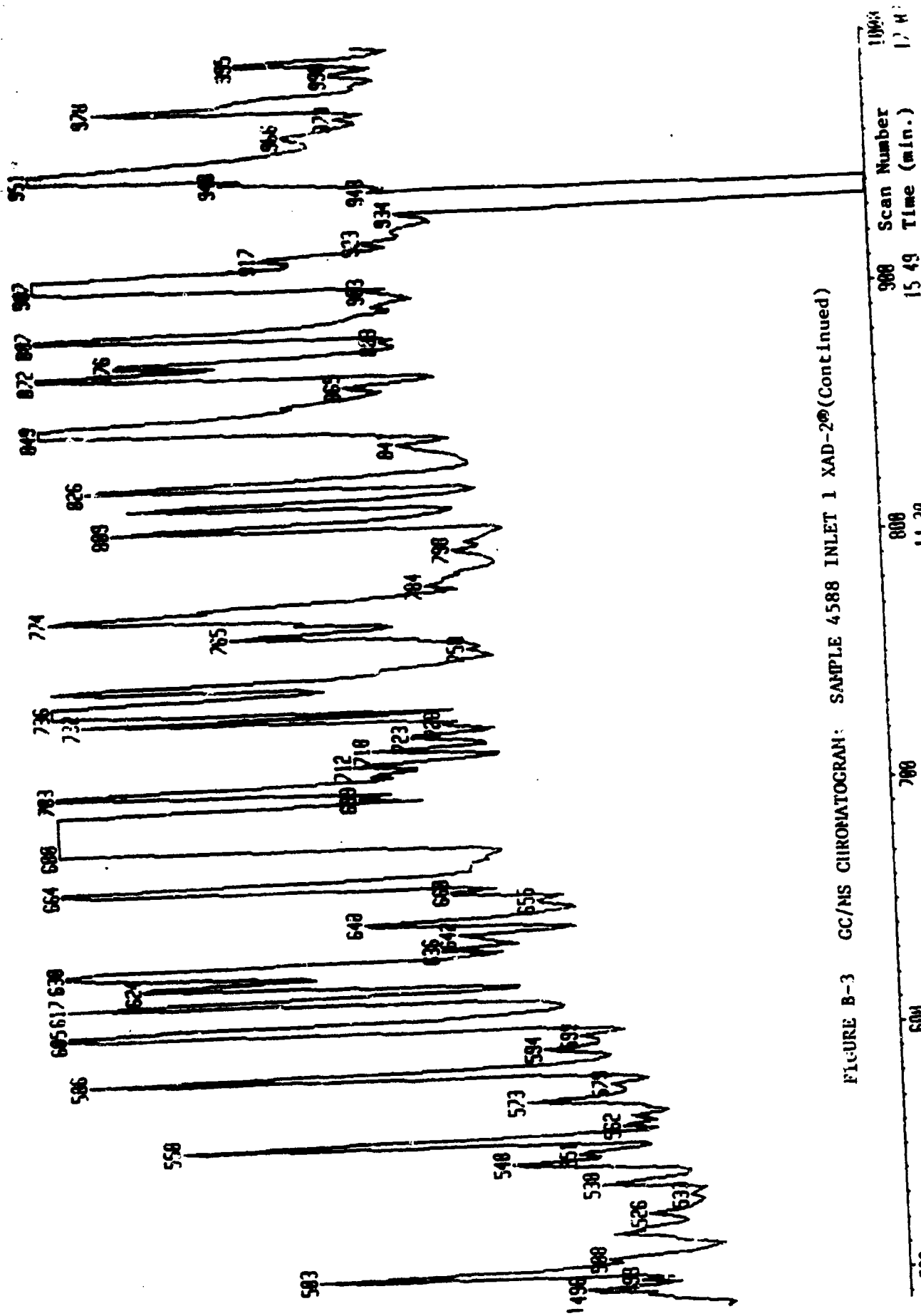
Source: Arthur D. Little, Inc.

▲ Arthur D. Little, Inc.
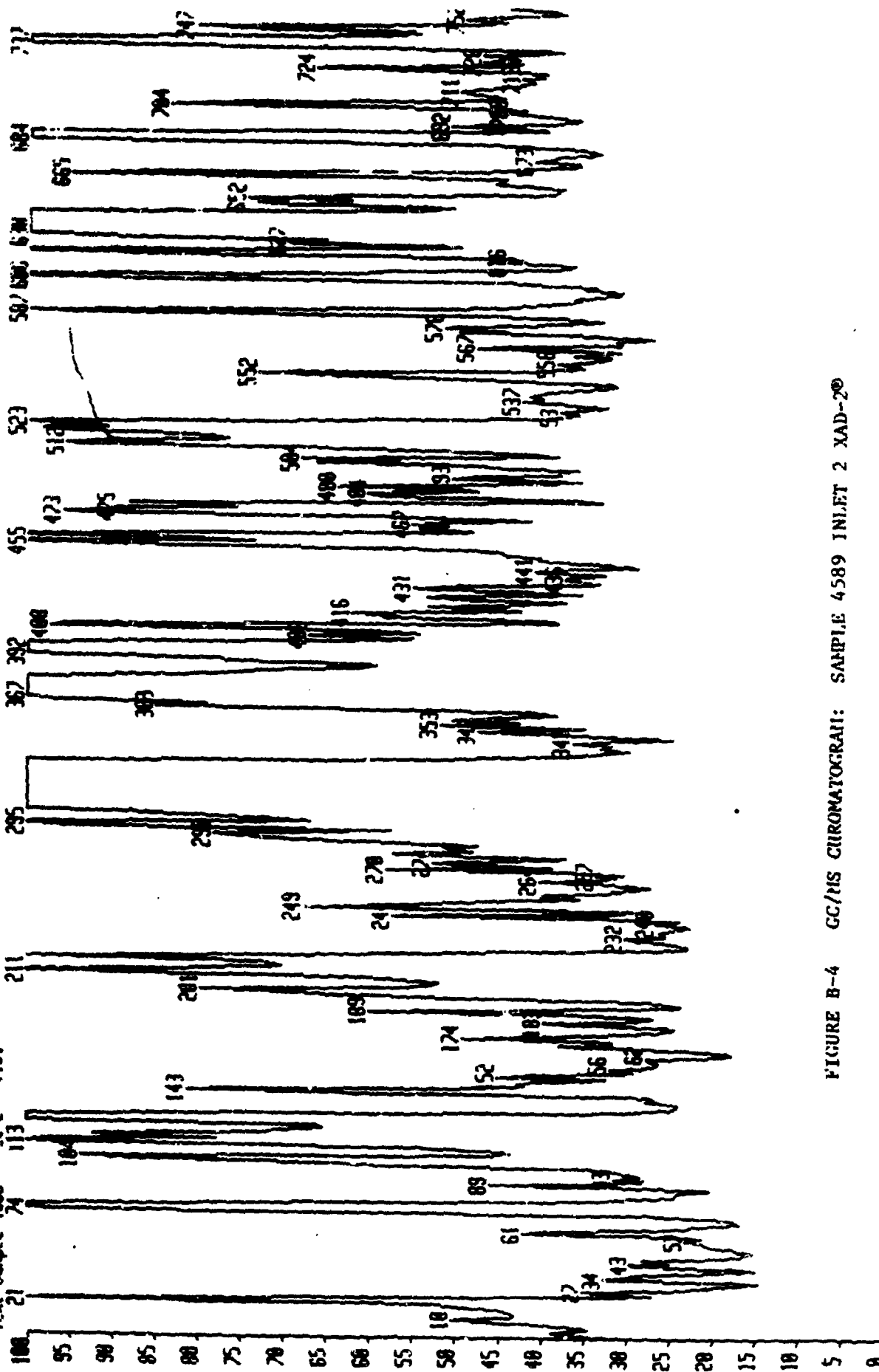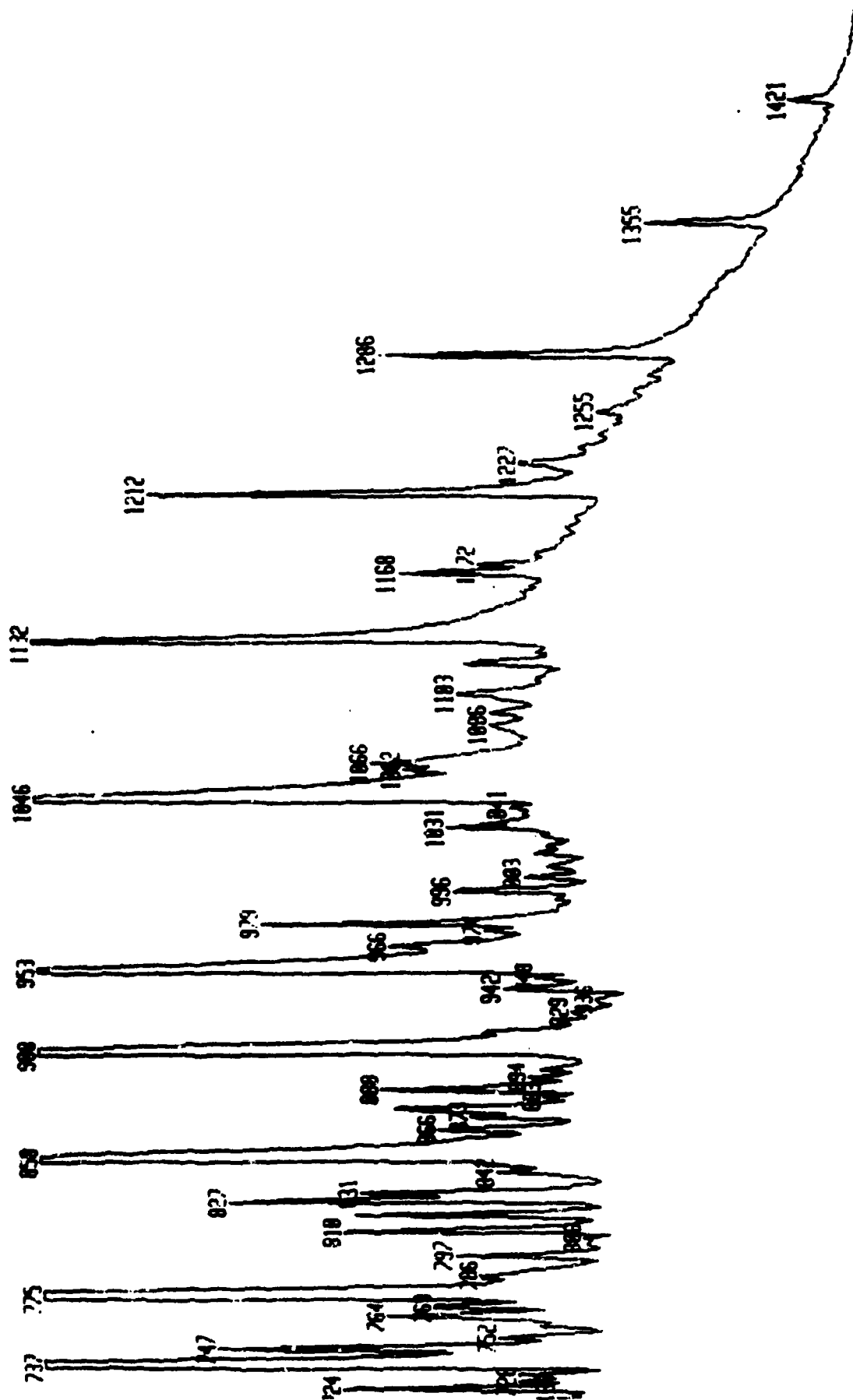
Chromatogram Identifiers: A :TIC
Text:Sample 4591 Field Blank + I.S. (NM2)
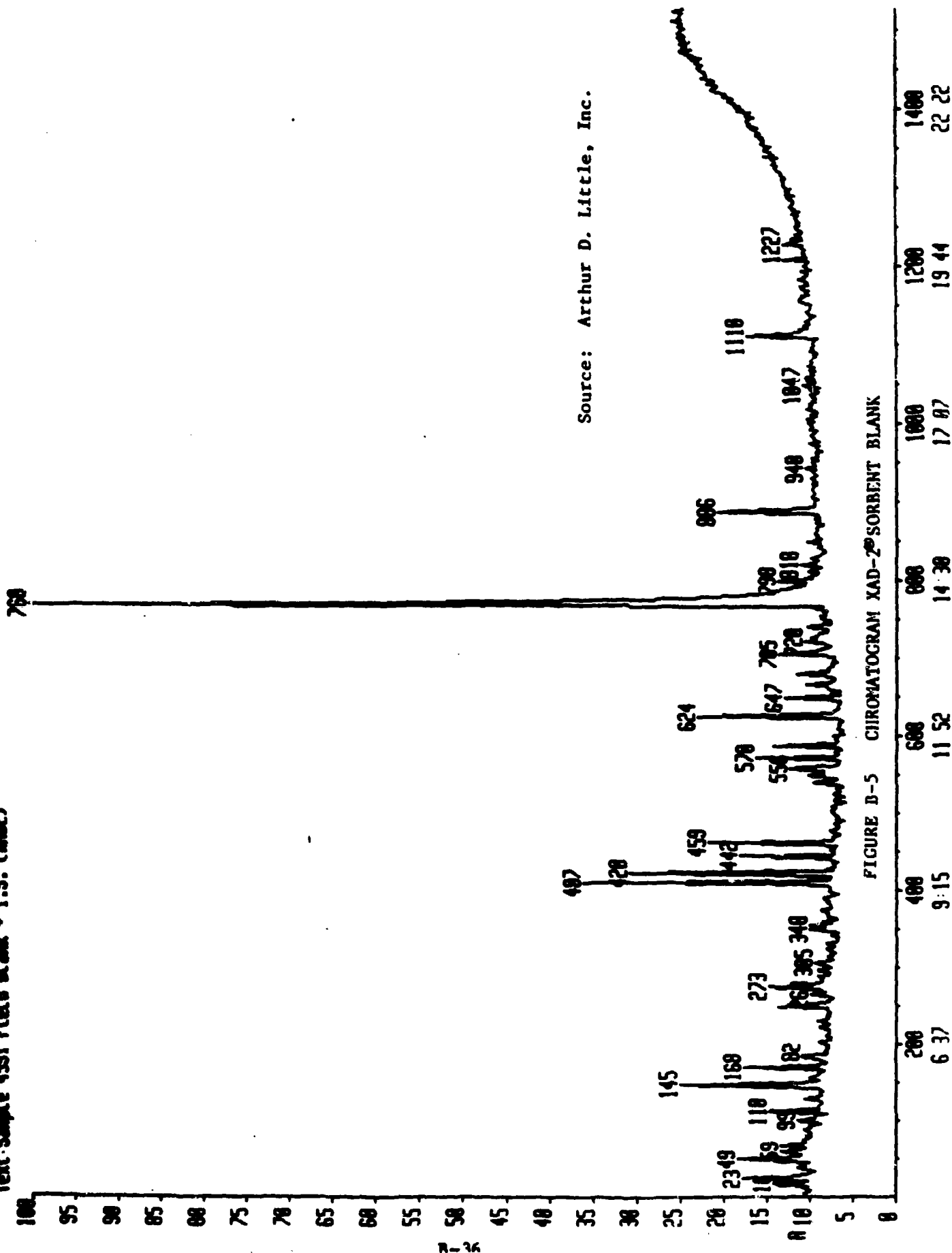
Source: Arthur D. Little, Inc.

FIGURE D-5   CHROMATOGRAM XAD-2® SORBENT BLANK

VII. **GC/MS Analysis of Concrete Block Core Extract after Decontamination**

In addition to analyzing the concentrated extract from the decontaminated concrete block core Sample No. 4628 (Table B-16), a blank concrete core extract and an acetonitrile solvent blank were also concentrated one hundred fold. The results for these analyses are given in Table B-17 and B-18, respectively.

Table B-19 is a listing of compounds which were detected only in the decontaminated concrete core extract. The mass detected and the concentration in $\mu g/g$ are given. All compounds identified are an order of magnitude lower in concentration than the concentration at which class 2 certification was received (10 ng on column).

The first analysis of the concrete core extract identified 5 compounds which were not detected in the second analysis of the extract. These compounds are listed below:

Scan Number	Tentative Identification
714	Phenol, 2,6-Bis (1,1-Dimethylethyl)-4-Methyl-
783	Long Chain Branched Hydrocarbon
879	Naphthoquinone Derivative
967	Phenol, 2,6-Bis (1,1-Dimethylethyl)-4-Ethyl-
1037	Naphthoquinone Derivative

Since the first analysis of the extract did not include quality control blank samples, it is unknown whether the above are associated with blank contamination problems. Therefore, the above compounds have been excluded from the summaries of data given in Tables B-16 and B-19.

TABLE B-16

COMPOUNDS IDENTIFIED IN CONCRETE CORE EXTRACT 4628

Scan	Tentative Identification
205	Benzene Methanol
210	Dichlorobenzene
304	Long Chain Alcohol
391	Naphthalene
425	Long Chain Alcohol
587	Propanoic Acid, Methyl, Dimethyl (Hydroxy-Methyl Ethyl) Propyl Ester
606	Propanoic Acid, Methyl, Hydroxy-Trimethyl Pentyl Ester
630	Dinitrobenzene
654	C14 H30 Alkane
698	2,5 Cyclohexadiene 1,4-Dione 2,6 Bis (1,1 Dimethyl Ethyl)
786	Bis (Dimethyl Propyl) Benzenediol
797	Phthalate
812	Unknown
824	Propanoic Acid, 2-Methyl,1-T-Butyl-2-Methyl, 1,3-Propanediyl Ester (C16 H30 04)
831	Trinitrobenzene
842	Long Chain Alkane
862	Trinitrotoluene
887	C16 H32 Alkene
928	Heptadecane
943	Long Chain Alkane
963	Phenanthrene
969	Internal Standard (D10 Anthracene)
1010	Octadecane
1018	Long Chain Acid (Possibly C14 H28 02)
1030	Phthalate
1048	Dinitrobenzamine (?)
1059	Long Chain Alcohol
1100	Phthalate
1156	Long Chain Alcohol
1177	Pyrene or Fluoranthene
1209	Long Chain Alkene
1213	Pyrene or Fluoranthene
1219	Phenyl Naphthalene
1232	Heneicosane
1257	Long Chain Amide
1300	Long Chain Alkane
1360	Phthalate
1365	Long Chain Alkane
1375	Long Chain Amide
1379	Long Chain Amide
1393	Long Chain Amide

COMPOUNDS IDENTIFIED IN CONCRETE CORE EXTRACT 4628

Scan	Tentative Identification
1405	Ethanol, 2-Butoxy-, Phosphate (?)
1428	Long Chain Alkane
1432	Chrysene or Triphenylene or Naphthacene
1471	Silylated Material
1488	Long Chain Alkane
1493	Phthalate
1502	Long Chain Amide
1546	Long Chain Alkane
1556	Silylated Material
1621	Long Chain Amide
1636	Silylated Material
1654	Benzo Pyrene or Benzofluoranthene
1663	Long Chain Branched Alkane
1796	Silylated Material
1894	Silylated Material

Source: Arthur D. Little, Inc.

TABLE B-17

COMPOUNDS IDENTIFIED IN CONCRETE CORE BLANK CONCENTRATE

Scan	Tentative Identification
34	Butoxy Ethanol
60	Methyl Pentanediol
81	Methoxy Ethoxy Ethanol
209	Benzene Methanol
376	Benzoic Acid
396	Naphthalene
403	Butoxy Ethoxy Ethanol
591	Propanoic Acid, Methyl, Dimethyl (Hydroxy-Methyl Ethyl) Propyl Ester
611	Propanoic Acid, Methyl, Hydroxy-Trimethyl Pentyl Ester
659	C14 H30 Alkane
703	2,5 Cyclohexandiene 1,4-Dione 2,6 Bis (1,1 Dimethyl Ethyl)
711	Substituted Methoxy Phenol (possibly C14 H24 O2)
791	Bis (Dimethyl Propyl)Benzenediol
797	Phenol,2,6-Bis(1,1-Dimethyl)-4-Ethyl-
801	Benzene Dicarboxylic Acid Diethyl Ester
828	Propanoic Acid, 2-Methyl,1-T-Butyl-2-Methyl, 1,3-Propanediyl Ester (C16 H30 O4)
846	Hexadecane
867	Trinitrotoluene
892	Long Chain Alkane
932	Heptadecane
941	C17 H34 Long Chain Alkane
973	Internal Standard (D10 Anthracene) - 5 ppm
1014	Octadecane
1023	Long Chain Acid (possibly C14 H28 O2)
1034	Phthalate
1092	Nonadecane
1105	Phthalate
1145	Sulfur (S8)
1166	Dodecane
1181	Pyrene or Fluoranthene
1237	Heneicosane
1264	Long Chain Alkane
1288	Long Chain Alcohol
1316	Long Chain Alcohol
1363	Phthalate
1383	Long Chain Amide
1394	Long Chain Alkane
1422	Dicyclohexyl Alkane
1455	Long Chain Alkane
1484	Cyclohexyl Alkane

COMPOUNDS IDENTIFIED IN CONCRETE CORE BLANK CONCENTRATE

Scan	Tentative Identification
1497	Phthalate
1507	Long Chain Amide
1625	Long Chain Amide
1640	Silylated Material
1667	Long Chain Branched Alkene
1716	Silylated Material
1800	Silylated Material
1898	Silylated Material

Source: Arthur D. Little, Inc.

TABLE B-18

COMPOUNDS IDENTIFIED IN ACETONITRILE CONCENTRATE

Scan	Tentative Identification
567	Silylated Material
604	Propanoic Acid, Methyl, Hydroxy-Trimethyl Pentyl Ester
795	Phthalate
821	Silylated Material
967	Internal Standard (D10 Anthracene)
1098	Phthalate
1358	Phthalate
1661	Long Chain Branched Alkene

Source: Arthur D. Little, Inc.

COMPOUNDS UNIQUE TO CONCRETE CORE 4628

Scan	Compound	Mass on Column (ng)	Concentration (ug/g block)
210	Dichlorobenzene	<1[a]	<0.01
304	Long Chain Alcohol	<1	<0.01
425	Long Chain Alcohol	<1	<0.01
630	Dinitrobenzene	<<1[b]	<<0.01
812	Unknown	1	0.01
831	Trinitrobenzene	1	0.01
963	Phenanthrene	<1	<0.01
1048	Dinitrobenzamine?	<1	<0.01
1059	Long Chain Alcohol	<1	<0.01
1156	Long Chain Alcohol	2	0.02
1209	Long Chain Alkene	1	0.01
1213	Pyrene or Fluoranthene	<1	<0.01
1219	Phenyl Naphthalene	<1	<0.01
1257	Long Chain Amide	<1	<0.01
1300	Long Chain Alkane	1	0.01
1375	Long Chain Amide	1	0.01
1379	Long Chain Amide	1	0.01
1393	Long Chain Amide	1	0.01
1405	Ethanol,2 Butoxy,Phosphate?	1	0.01
1432	Chrysene or Triphenylene or Naphthacene	<1	<0.01
1471	Silylated Material	<1	<0.01
1488	Long Chain Alkane	1	0.01
1546	Long Chain Alkane	<1	<0.01
1556	Silylated Material	<1	<0.01
1654	Benzopyrene or Benzofluoranthene	1	0.01

[a] < Less than
[b] << Much less than

Source: Arthur D. Little, Inc.

⚠ Arthur D. Little, Inc.

APPENDIX C

LISTING OF BEND AND COMPRESSION TEST RESULTS

FOR CONCRETE BLOCK AND CONCRETE SAMPLES

APPENDIX C

The following tables list all of the bend and compression
test results for the two pilot tests for concrete block
and concrete samples.

Note: For all samples:

        b and d = specimen cross section dimensions (cm)

        Load   = applied load (kg)

C-1

TABLE C-1

BUILDING 4L-5 HEATED CONCRETE BLOCK BEND TEST RESULTS

FIRST PILOT TEST

Sample No.	b (cm)	d (cm)	Load (Kg)	Bend Strength (psi)	Maximum Block Temp. (°F)
I-I-A	2.140	2.010	20.0	310	666
I-1-B	2.030	1.940	28.0	492	666
I-1-C	2.040	2.000	23.5	386	666
I-1-D	2.120	2.060	30.5	455	666
I-6-A	2.070	2.000	21.5	348	656
I-6-B	1.965	1.995	23.2	398	656
I-6-C	1.950	1.840	30.0	609	656
I-6-D	2.195	2.130	21.5	290	656
I-8-A	1.970	1.980	25.8	448	661
I-8-B	2.100	1.870	---	---	661
I-8-C	2.000	1.890	22.1	415	661
I-8-D	1.970	1.520	17.0	501	661
O-5-A	2.360	2.100	---	---	414
O-5-B	2.020	1.965	34.6	595	414
O-5-C	1.960	1.900	---	---	414
O-5-D	1.925	1.940	19.4	359	414
O-10-A	2.080	1.755	14.5	304	518
O-10-B	2.030	1.715	16.0	356	518
O-10-C	2.130	1.785	11.0	217	518
O-10-D	1.965	1.455	11.5	371	518
O-17-A	2.225	2.100	40.7	556	350
O-17-B	1.975	1.915	35.0	648	350
O-17-C	1.980	1.940	24.3	437	350
O-17-D	1.850	1.710	16.5	409	350

SECOND PILOT TEST

Sample No.	b (cm)	d (cm)	Load (Kg)	Bend Strength (psi)	Maximum Block Temp. (°F)
1-I-9	2.085	2.065	27.5	415	513
2-I-9	1.950	2.020	26.7	450	513
3-I-11	2.130	2.025	36.5	561	532
4-I-11	1.960	1.960	32.0	570	532
5-I-13	2.010	1.960	22.0	382	574
6-I-13	1.970	1.945	30.5	549	574
7-O-10	2.315	2.050	46.0	634	321
8-O-10	2.310	2.000	66.0	958	321
9-O-12	2.245	2.022	33.0	482	393
10-O-12	2.240	2.000	25.5	362	393
11-O-14	2.050	2.050	37.5	584	445
12-O-14	2.050	1.965	36.5	618	445

Source: Arthur D. Little, Inc.

Arthur D. Little, Inc.

TABLE C-2

BUILDING 4L-5 UNHEATED CONCRETE BLOCK BEND TEST RESULTS

Sample No.	b (cm)	d (cm)	Load (Kg)	Bend Strength (psi)	Comments
W-1	1.905	1.715	49.5	963	as received
W-1-a	2.150	1.850	73.5	1340	as received
W-2	2.005	1.915	65.0	1186	as received
W-2-A	2.015	1.750	37.0	804	as received
W-3	2.140	1.945	57.5	953	as received
W-3-A	1.995	1.820	52.4	1064	as received
W-4	2.000	1.750	33.8	740	as received
W-4-A	1.905	1.794	30.1	657	as received
CAAP-A	2.100	1.900	60.5	1070	as received
CAAP-B	1.99	1.975	54.5	<u>942</u>	as received

Average Bend Strength (psi)   972

Source: Arthur D. Little, Inc.

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TABLE C-3

COMPRESSION TEST RESULTS FOR NEW CONCRETE BLOCK

Sample No.	b (cm)	d (cm)	Load (Kg)	Compressive Strength (psi)	Comments	Heating Cycle
1-C-400-A	3.200	3.150	2175	3063	ht. @ 400°F	
2-C-400-B	3.200	3.230	1850	2540	ht. @ 400°F	
3-C-400-B	3.170	3.210	2525	3522	ht. @ 400°F	5 hrs 400°F
4-C-400-C	3.200	3.160	2460	3453	ht. @ 400°F	
5-C-400-D	3.170	3.170	2475	3496	ht. @ 400°F	
6-C-500-A	3.090	3.160	2350	3416	ht. @ 500°F	
7-C-500-B	3.140	3.190	1830	2593	ht. @ 500°F	1 hr 400°F
8-C-500-C	3.200	3.170	2240	3134	ht. @ 500°F	3 hrs 500°F
9-C-500-C	3.210	3.160	1925	2694	ht. @ 500°F	
10-C-500-D	3.180	3.230	2540	3510	ht. @ 500°F	
11-C-600-A	3.170	3.140	2040	2909	ht. @ 600°F	
12-C-600-B	3.290	3.240	1875	2497	ht. @ 600°F	1 hr 400°F
13-C-600-C	3.130	3.200	2025	2870	ht. @ 600°F	1 hr 500°F
14-C-600-D	3.180	3.210	2075	2885	ht. @ 600°F	2 hrs 600°F
15-C-600-D	3.210	3.200	2400	3316	ht. @ 600°F	
16-N-A	3.200	3.140	2060	2910	not ht.	
17-N-A	3.120	3.210	2050	2905	not ht.	
18-N-B	3.230	3.200	2710	3721	not ht.	
19-N-C	3.200	3.160	2575	3614	not ht.	
20-N-D	3.220	3.240	2830	3850	not ht.	

Average Strength (psi)					
	3400	not ht.
	3215	ht. @ 400°F
	3069	ht. @ 500°F
	2895	ht. @ 600°F

Note: ht. — heat treated
       not ht. — not heat treated

Source: Arthur D. Little, Inc.

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TABLE C-4

BEND TEST RESULTS FOR NEW CONCRETE BLOCK

Sample No.	b (cm)	d (cm)	Load (Kg)	Bend Strength (psi)	Comments
1-N-400-A	2.500	2.550	70.5	582	ht. @ 400°F
2-N-400-B	2.600	2.4800	67.5	566	ht. @ 400°F
3-N-400-C	2.620	2.600	60.0	454	ht. @ 400°F
4-N-400-D	2.530	2.520	62.0	518	ht. @ 400°F
5-N-400-A	2.560	2.570	61.1	485	ht. @ 400°F
6-N-500-A	2.500	2.550	41.0	338	ht. @ 500°F
7-N-500-B	2.530	2.500	52.0	441	ht. @ 500°F
8-N-500-C	2.560	2.580	41.0	323	ht. @ 500°F
9-N-500-C	2.600	2.650	64.5	474	ht. @ 500°F
10-N-500-D	2.520	2.530	44.0	366	ht. @ 500°F
11-N- 0-A	2.540	2.600	54.5	426	ht. @ 600°F
12-N-600-B	2.500	2.510	43.5	370	ht. @ 600°F
13-N-600-C	2.610	2.610	50.5	381	ht. @ 600°F
14-N-600-D	2.520	2.540	57.0	470	ht. @ 600°F
15-N-600-D	2.540	2.520	41.5	345	ht. @ 600°F
16-N-A	2.620	2.520	91.0	734	not ht.
17-N-B	2.490	2.660	104.0	792	not ht.
18-N-B	2.550	2.510	30.5	255	not ht.
19-N-C	2.680	2.600	79.0	585	not ht.
20-N-D	2.550	2.520	89.5	741	not ht.

		Average Strength (psi)			
				621	not ht.
				521	ht. @ 400°F
				388	ht. @ 500°F
				398	ht. @ 600°F

Note: ht. — heat treated
       not ht. — not heat treated

Source: Arthur D. Little, Inc.

Arthur D. Little, Inc.

TABLE C-5

BUILDING 4L-5 CONCRETE COMPRESSION TEST RESULTS

SECOND PILOT TEST

Sample No.	b (cm)	d (cm)	Load (Kg)	Strength (psi)	Comments
1-2A	2.930	2.820	1400	2405	heated floor 445°F avg.
1-2A	2.330	2.440	950	2372	heated floor 445°F avg.
1-2	2.660	2.680	1625	3235	floor unheated
1-2	2.620	2.700	1650	3311	floor unheated
1-2	2.500	2.660	1325	2828	floor unheated
0-18-C1	2.730	2.670	1650	3213	lintel heated 500°F max.
0-18-C2	2.670	2.620	2075	4210	lintel heated 500°F max.
I-17-D1	2.950	2.830	1975	3358	lintel heated 580°F max.
I-17-D2	2.810	2.880	1600	2806	lintel heated 580°F max.
E1	2.640	2.560	2150	4515	lintel unheated
E2	2.680	2.640	2900	5818	lintel unheated

Source: Arthur D. Little, Inc.

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